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The laser-induced fluorescence from Dy:YAG has been employed for the measurement of temperature on reacting and nonreacting surfaces. Point, line, and two-dimensional mapping of the surface temperature has been demonstrated utilizing the tripled output (355 nm) of a Nd:YAG laser. The temporal and spatial distribution of the temperature was recorded with a two-dimensional intensified imaging system. The technique has also been utilized for the measurement of the thermal depth profile of a plastic material undergoing rapid heating by a CO₂ laser. A fiber-optic temperature probe was developed. Fluorescent crystals bonded to the fiber-optic tip with a ceramic adhesive provide a 300 - 1100 K temperature range for the probe.

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SURFACE THERMOMETRY OF ENERGETIC MATERIALS
BY LASER-INDUCED FLUORESCENCE

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Covering the Period February 1987 through August 1989

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Foreword

This report was prepared by Michael E. Post and Larry P. Goss, Ph.D., Principal Investigator, and covers work performed under AFOSR Contract No. F49620-87-C-0040 during the period February 1987 through August 1989. The AFOSR contract monitor during this period was Julian M. Tishkoff, Ph.D.

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Section I

INTRODUCTION

This report presents the results of an experimental investigation into the use of Laser-Induced Fluorescence (LIF) as a diagnostic tool for remote sensing of the temperature of reacting and nonreacting surfaces. The goals of this effort were 1) to extend LIF techniques to the measurement of two-dimensional surface temperatures, 2) to develop a probe for measuring thermal depth profiles for reacting materials, and 3) to apply fluorescence temperature techniques to the study of nonmetallic materials.

Efforts made during this program toward achieving these goals are summarized in this report.

Overview of Laser-Induced Fluorescence

As a result of previous research, LIF was selected as the technique for remote sensing of the temperature of reacting and nonreacting surfaces. An overview of the LIF techniques employed in the experimental investigations described in this report is presented here. Additional details on these LIF methods can be found in "Applications of Atomic Fluorescence to Measurement of Combustion Temperature in Solid Propellants" (AFOSR Final Report dated December 1986).¹

Laser-Induced fluorescence of thermographic phosphors involves embedding small 10 - 100 μm sized thermographic-phosphor crystals in the surface to be studied. The LIF emission from these phosphors can be used to determine the temperature of the crystal and, thus, the surrounding surface. An ideal crystal for this purpose was found to be Dy:YAG; this crystal was employed for all the studies described in this report.

The temperature sensitivity of Dy:YAG crystals results from a "thermalization" process. When two energy levels of a rare-earth ion in a crystal field are separated by $< 1000 \text{ cm}^{-1}$, the upper level typically will not fluoresce at low temperature due to extremely high multi-phonon

relaxation rates which act to quench the closely spaced levels. Thus, at low temperatures no population buildup occurs in the upper energy level and, therefore, no fluorescence is observed. As the temperature increases, the upper energy level becomes more populated and the fluorescence from this level increases. Thus, by monitoring the increase in fluorescence of the upper level relative to the lower level, the temperature can be determined.

A prime example of this behavior is found in trivalent dysprosium doped at 3% yttrium-aluminum-garnet (Dy:YAG). The simplified energy diagram of Dy:YAG is shown in Fig. 1. Absorbed laser light (at 355 nm) can excite the Dy^{+3} ion into a high-energy state which radiatively and non-radiatively decays to the F-level. This level undergoes a fast thermal equilibrium which pumps a portion of its population into the nearby G-level. Fluorescence is then observed from both states. Figure 2 displays the fluorescence from both the F- and G-levels of Dy:YAG, including various Stark-shifted components, as a function of temperature. The intensity of the F-fluorescence level located at 496 nm is observed to remain relatively fixed as the temperature increases, while the intensity of the G-level component located at 467 nm increases dramatically with temperature. Because the 496-nm line is relatively insensitive to temperature, it can be used as an internal standard which allows the temperature to be determined from a ratio calculation rather than a more-difficult absolute measurement. The ratio of the 467- to 496-nm lines is shown in Fig. 3. A nonlinear curve observed for this ratio displays good temperature sensitivity up to 1800 K. The fluorescence from the F- and G-levels of Dy:YAG is extinguished at a temperature of ~ 1800 K due to the large phonon quenching rates at this high temperature. The temperature range of the Dy:YAG crystals is more than adequate for monitoring the surface temperature of energetic materials.

One of the major advantages of employing LIF thermographic phosphors is the possibility of extending the technique to multi-dimensional measurements through the use of line and/or two-dimensional detectors (cameras). Simultaneous monitoring of the F- and G-fluorescence levels in this manner permits determination of the surface temperature at a point, along a line, or over the entire surface. The experimental arrangement

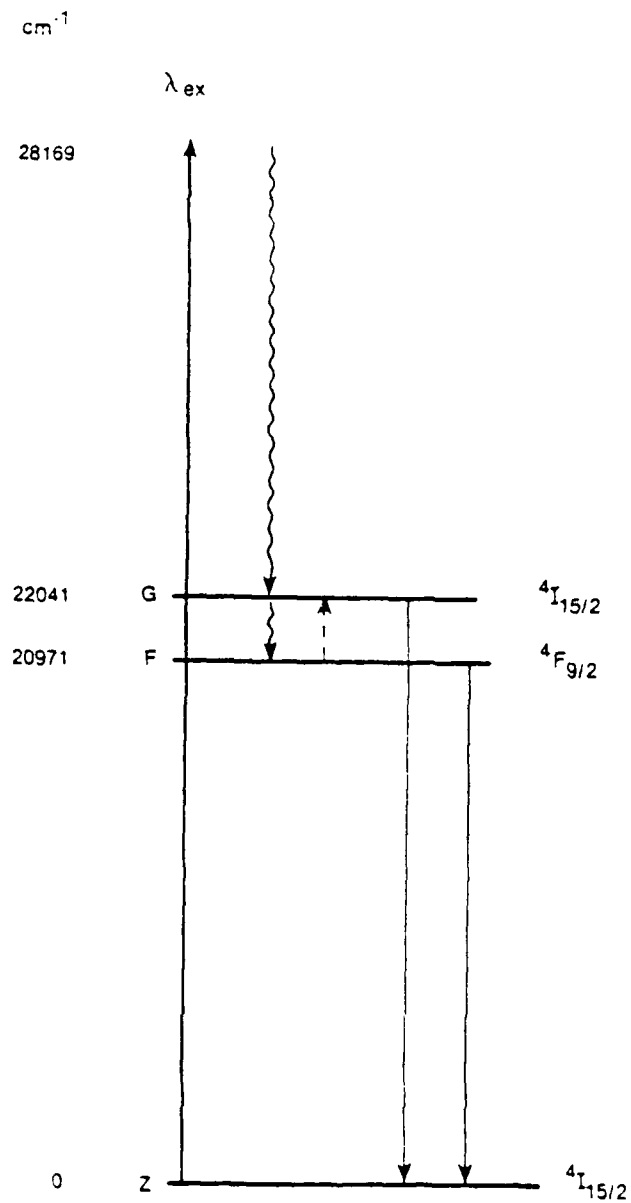


Figure 1. Simplified Energy Diagram of F- and G-Levels of Dy:YAG.

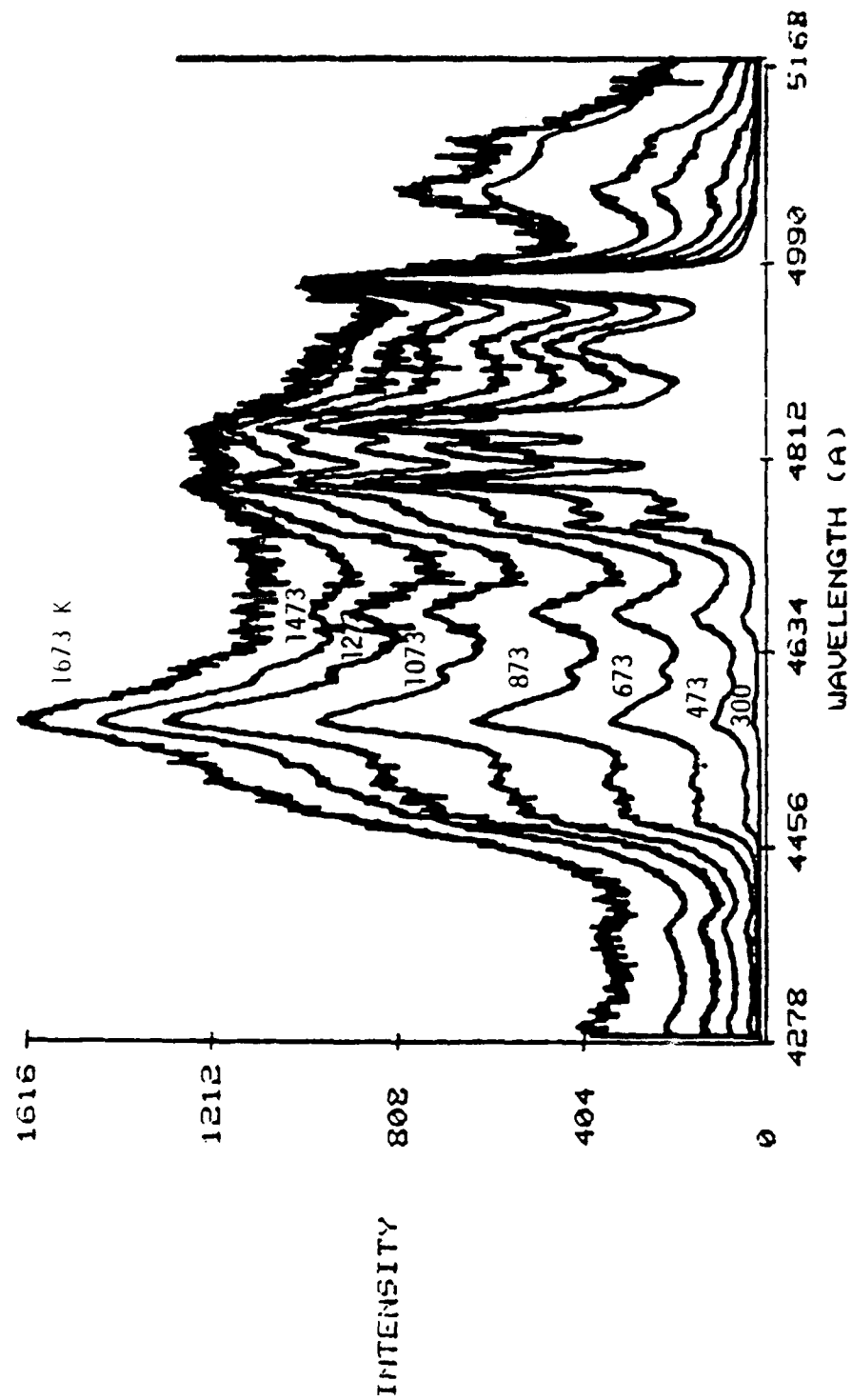


Figure 2. Fluorescence of F- and G-Levels of Dy:YAG as Function of Temperature.

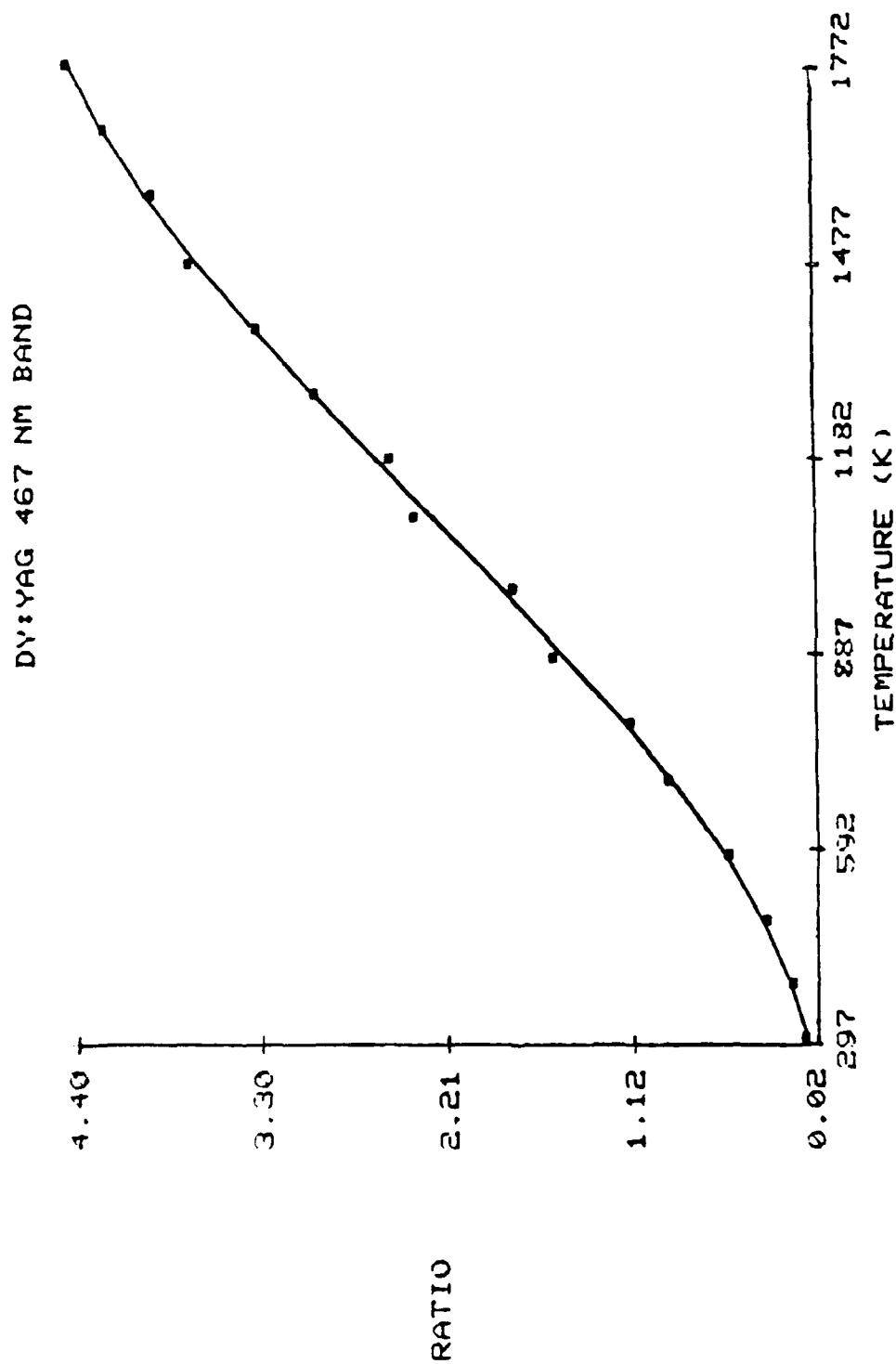


Figure 3. Variation of Relative Intensity of 467-nm G-Level Stark Component to 496-nm F-Level of Dy:YAG as Function of Temperature.

employed for surface thermographic imaging is shown in Fig. 4. The main component of the system is a linear or two-dimensional intensified camera which records the fluorescence from the surface under study. In the case of line measurements, the detector employed is a Tracor-Northern TN1710 1024-element diode array. For two-dimensional measurements the Tracor-Northern is replaced with an intensified CCD camera system manufactured by Photometrics, Ltd. In both cases the fluorescence from the surface is excited by the tripled output of a Quanta-Ray Nd:YAG laser. The intensified detector is gated with a 10- μ s pulse which is an aid in discriminating against unwanted background radiation from the surface. This feature is very important at elevated temperatures where the natural blackbody emission from the surface and surroundings can be particularly large. The fluorescence from the surface is collimated by an F/6 lens, split into two paths, and directed through 496- and 467-nm filters corresponding to the F- and G-fluorescence levels, respectively. The two paths are then imaged side by side on the linear or CCD camera. The video signal from the detector is digitized, stored in temporary memory, and subsequently sent to a mini-computer for analysis. Image analysis consists of ratioing the F- and G-fluorescence signals and converting the resulting relative intensity to a temperature with the aid of a calibration curve established using a platinum-coiled oven. The surface temperature can then be displayed as a function of time and/or space. The Tracor-Northern linear-array detector is capable of 30-Hz operation and, thus, has been employed for time-dependent temperature studies. Since the Photometrics CCD has a much lower data-acquisition rate, it has been employed for precise spatial measurements.

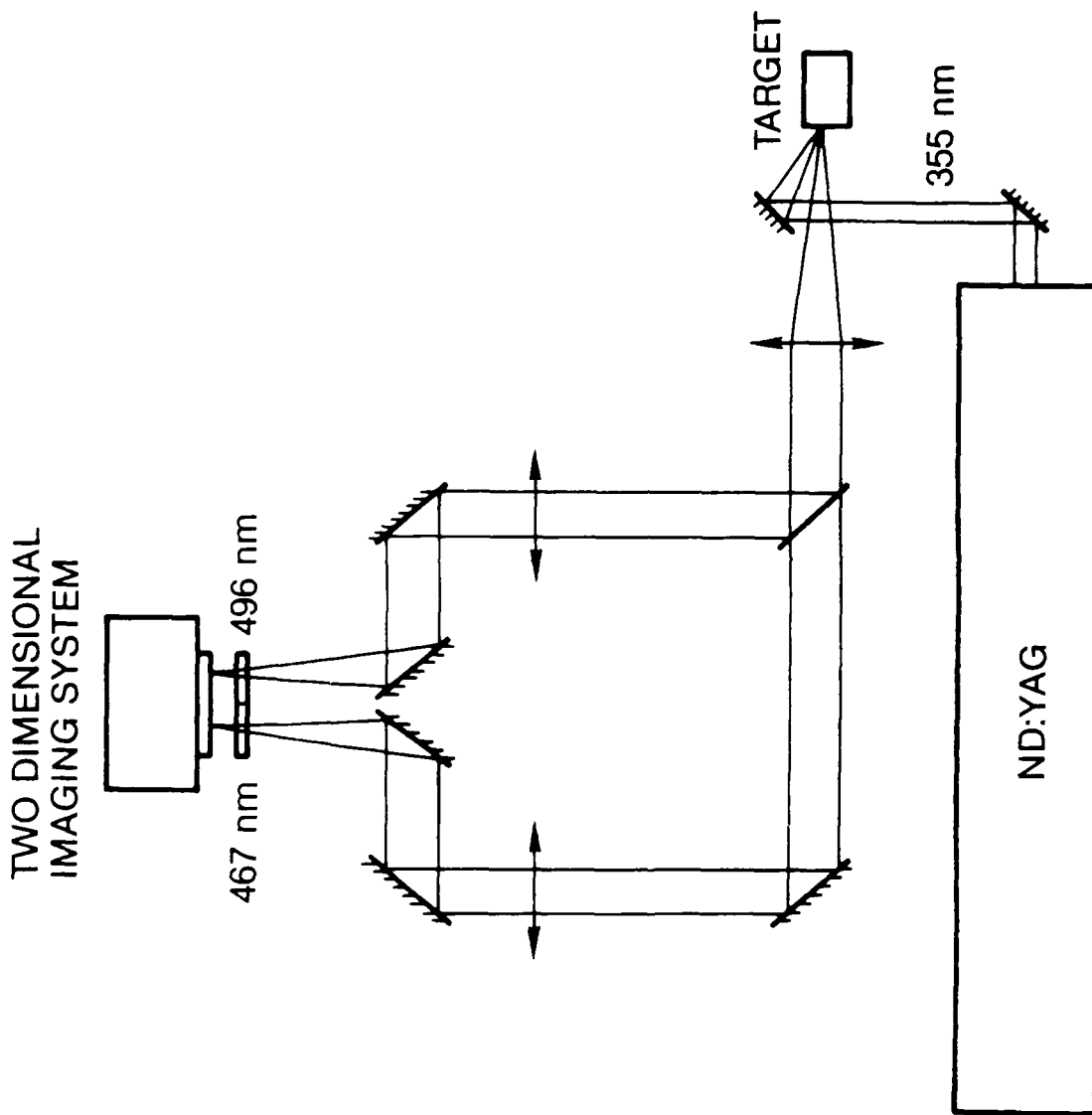


Figure 4. Experimental Arrangement Employed for Surface Thermographic Imaging.

Section II

LABORATORY STUDIES

Line and Two-Dimensional Surface Studies

The primary emphasis during the first year of this program was on line and two-dimensional surface studies. These are described in greater detail in "Surface Thermometry of Energetic Materials by Laser-Induced Fluorescence" (AFOSR Annual Technical Report dated April 1988).²

The linear-time-resolved imaging effort was concentrated on inert ceramic and plastic surfaces. The various heat sources used to increase the surface temperature of the test specimen were 1) embedded, electrically heated Nichrome wires and 2) CO₂ laser excitation.

Linear-time-resolved temperature measurements on a nonreacting surface were made using the experimental arrangement shown in Fig. 5(a). The surface consisted of small (< 50- μ m) Dy:YAG crystals implanted in a ceramic material in which a small Nichrome wire had been embedded. An electrical current was allowed to flow through the wire to act as a local heat source, and the fluorescence from the 467-nm G-Stark component was monitored by a linear multichannel detector along a line perpendicular to the wire filament. The fluorescence was recorded in a single 10-ns pulse and stored by the computer to obtain a time history of the temperature changes at the surface under heat-up and cool-down conditions [5(b-c)]. Under heat-up conditions [5(b)], the initial rise in temperature was centered at the embedded wire, with a gradual transfer of heat to the surrounding surface. After 22 s, the maximum temperature rise was observed in the area surrounding the embedded wire (400 K). The heat buildup beyond this point was slower, asymptotically approaching 420 K. The cooling of the surface after interruption of electrical current to the wire filament is shown in Fig. 5(c). The temperature decline is steep in the peak-temperature area but slows as the central temperature approaches that of the surrounding heated area.

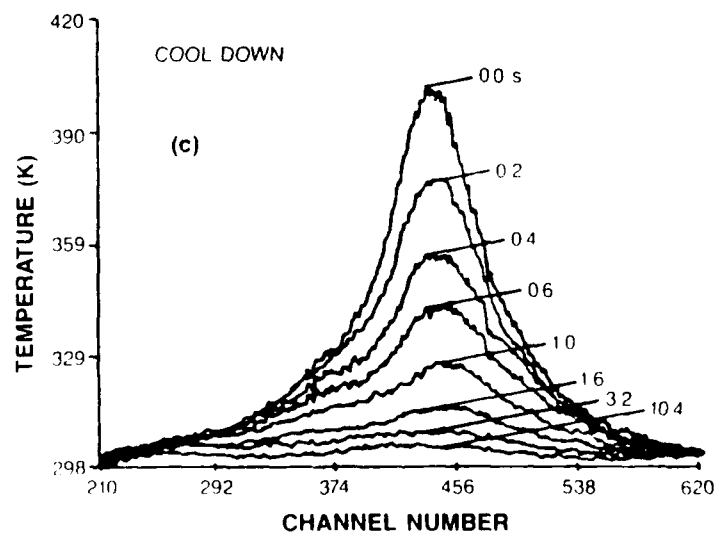
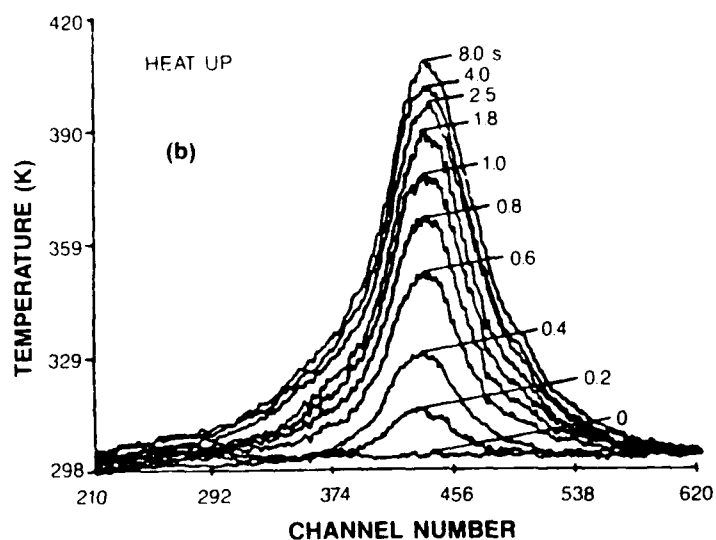
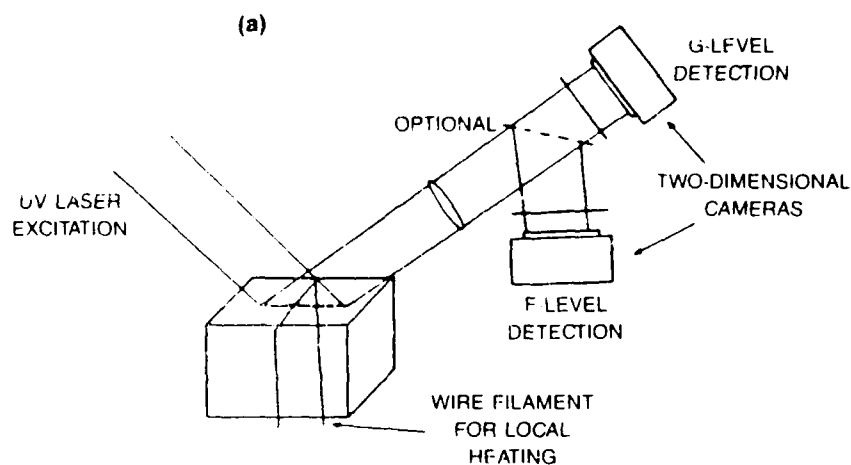


Figure 5. (a) Experimental Arrangement for Surface Thermometry Studies; (b), (c) Temporal Variation of Surface Temperature of Ceramic Material under Heat-Up and Cool-Down Conditions, Respectively.

Figure 6 depicts the change in surface temperature along a line perpendicular to the embedded wire. The temperature is displayed as a function of time as electrical current is allowed to flow through the Nichrome wire and heat the surface. After ~ 4.5 s, the electrical current is discontinued and the surface allowed to cool. From this figure the lateral spread in temperature as well as the cooling decay time can be determined.

Next, the ceramic surface was heated at a much higher rate by a CO_2 laser. The color contour of the temperature along a line intercepting the focal spot of the laser is shown in Fig. 7. The risetime in temperature is much faster with the CO_2 excitation than in the Nichrome-wire study. The temperature at the focal spot is also much higher than in the resistance-heating experiment. The CO_2 laser excitation began at ~ 4 s and ended at 10.5 s. The temperature then abruptly decayed, eventually reaching room temperature.

To demonstrate the thermometric technique on a surface similar to that of an energetic material, a thermal-setting plastic supplied by Buehler, Ltd., was employed. The Dy:YAG crystals were mixed in a 1:7 weight ratio with the plastic which was heated under pressure to form the test specimen. The plastic surface erodes as it is heated above 400 K; thus, the interdispersed crystals are continuously replenished at the surface. Therefore, no loss in the fluorescence signal is observed as the surface erodes; and the surface-temperature profile can be obtained, even on reactive surfaces.

The results of the first experiment with a plastic target are shown in Fig. 8. The surface of the plastic in this case was being heated with a propane torch which was covering the entire measurement area. The torch was applied to the surface at approximately the 4.5-s mark, and the temperature at the surface increased to ~ 550 K.

Next, the CO_2 laser was used to heat the plastic surface, and the results are shown in Fig. 9. The color contour shows the much higher surface temperature which was achieved by CO_2 excitation, with a maximum temperature over 700 K. The temperature decay after termination of the

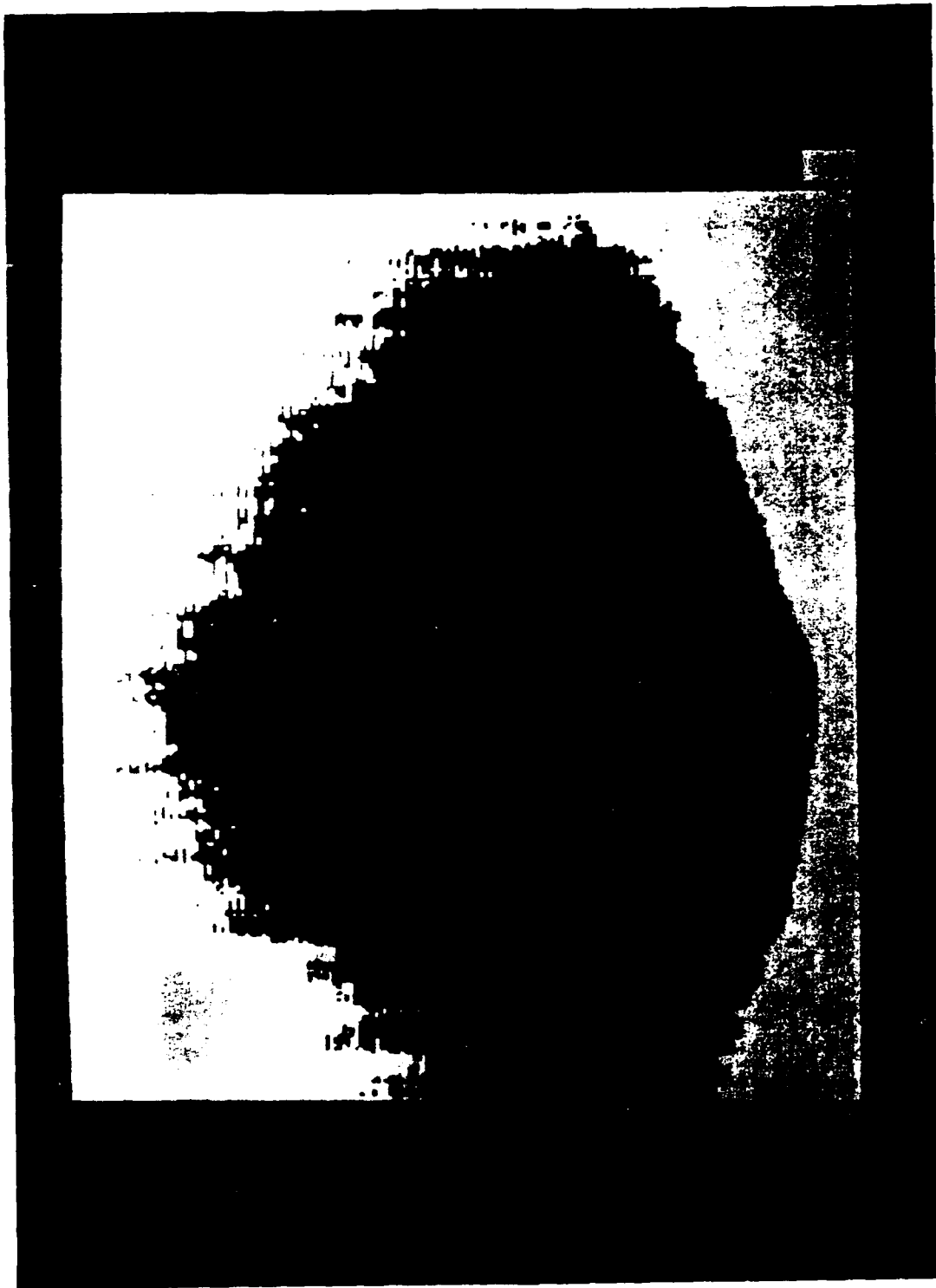


Figure 6. Color Contour Depicting Change in Surface Temperature Along
Line Located Perpendicular to Embedded Wire.

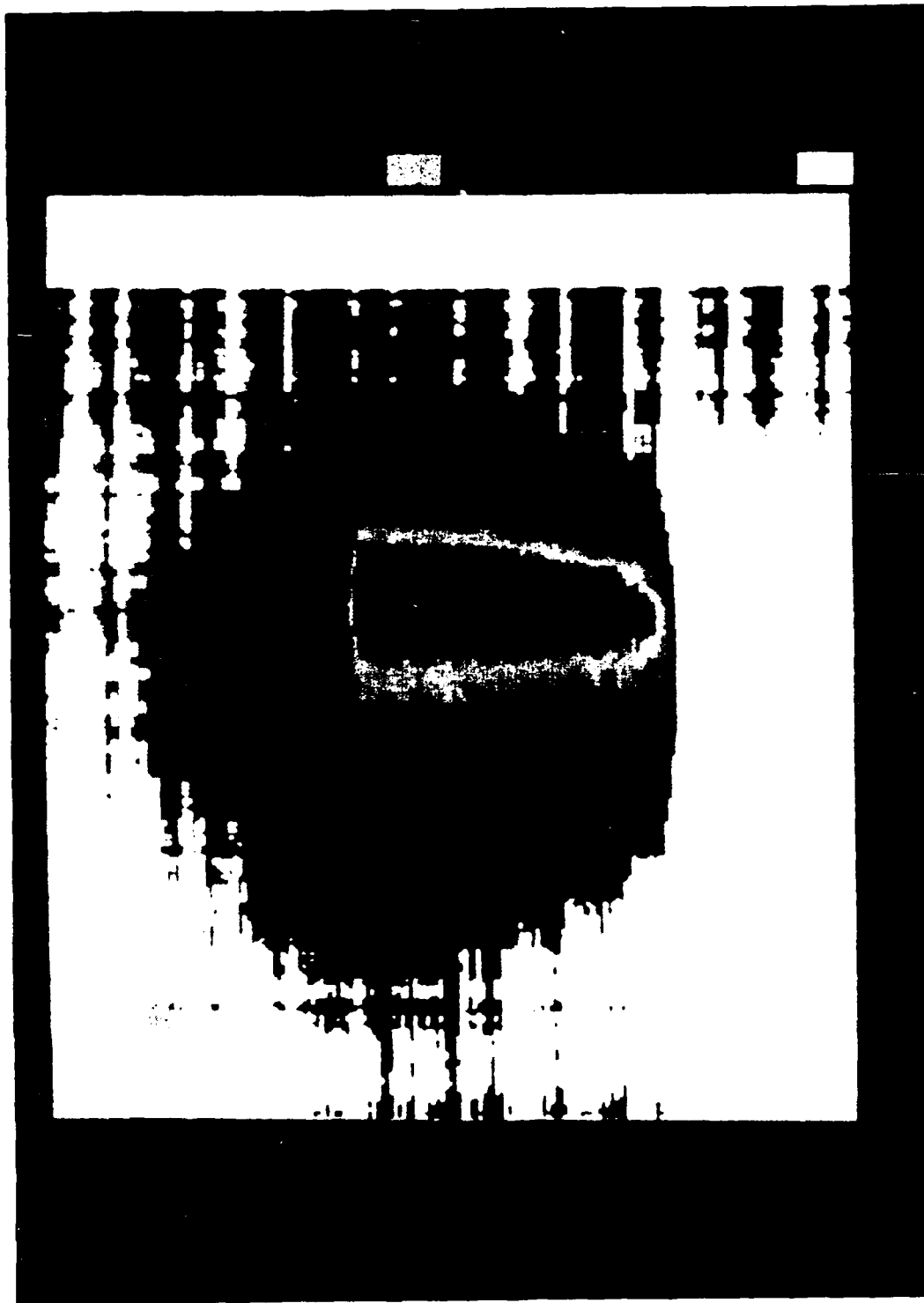


Figure 7. Color Contour of Temperature Along Line Intercepting Focal Spot of Laser.

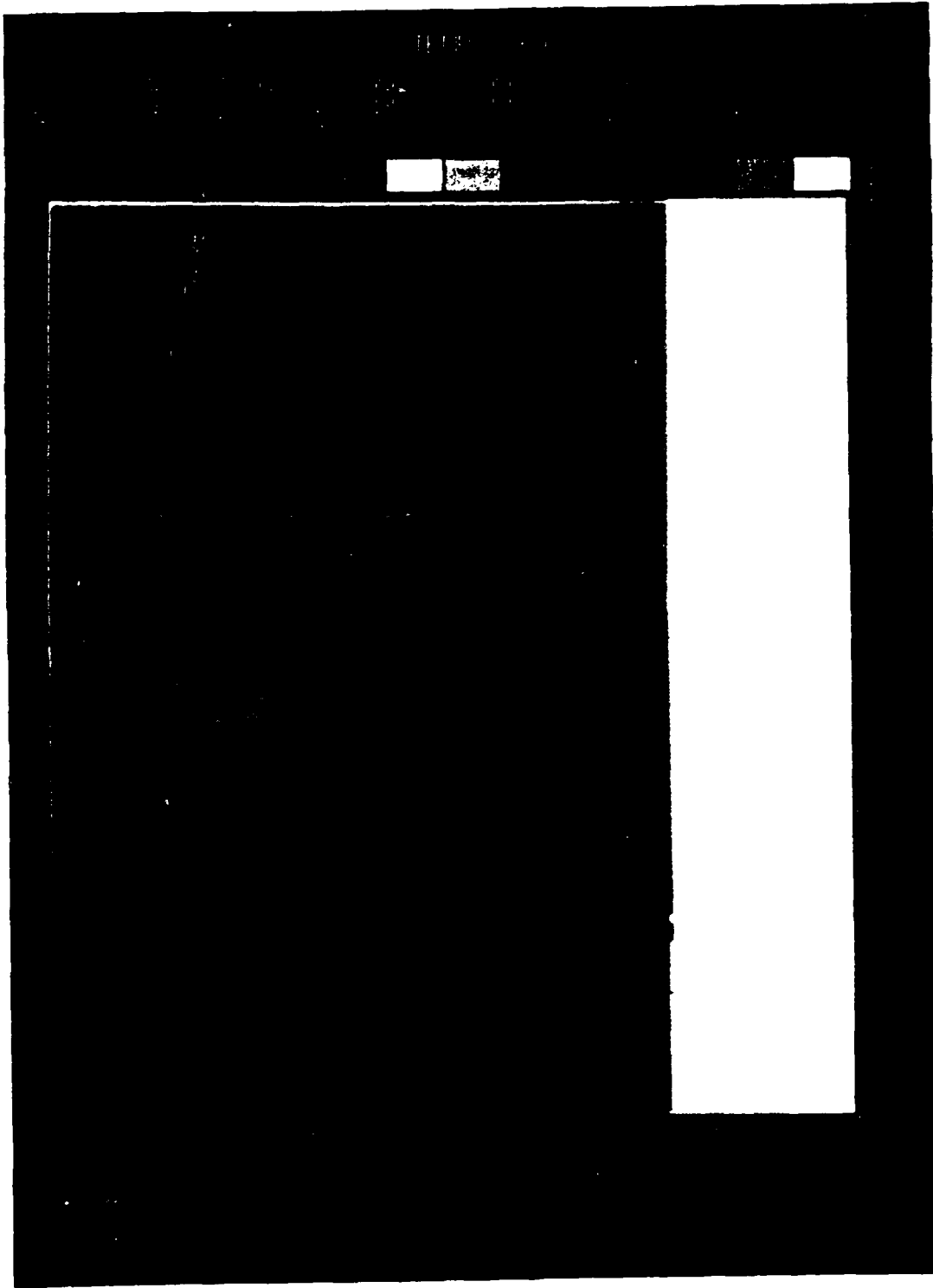


Figure 8. Color Contour Showing Results of Heating Plastic Surface with Propane Torch.

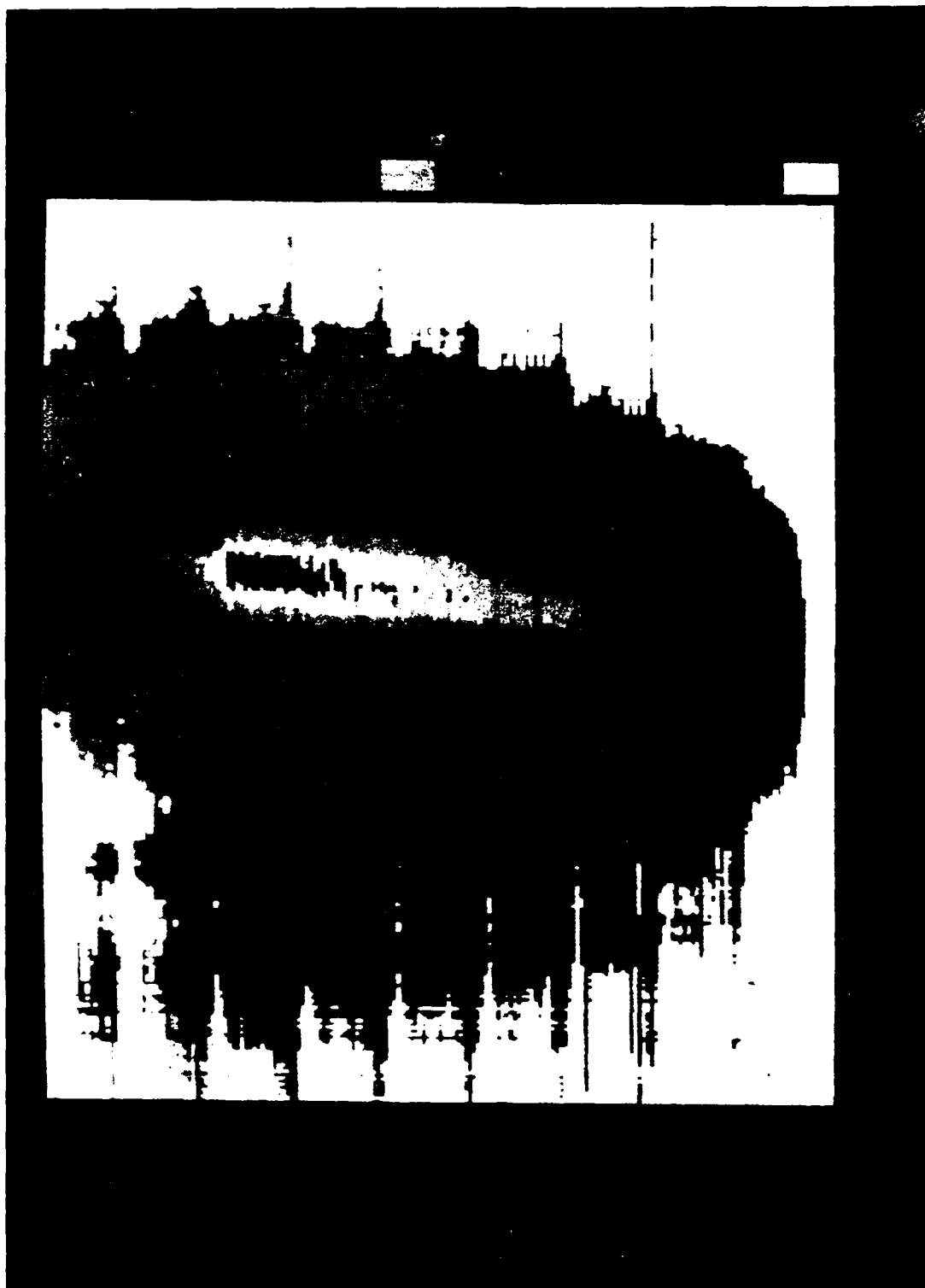


Figure 9. Color Contour Showing Results of Heating Plastic Surface
with CO₂ Laser.

CO₂ excitation is shown beginning at ~ 15.5 s. The plastic reached a lower temperature than the ceramic. This is partially due to the energy used in eroding and melting the plastic.

Figure 10 shows the excitation of the plastic surface which was recorded over two heat-up and cool-down cycles. Under excitation by the CO₂ laser, the plastic surface is actually eroded, as evidenced in Fig. 10, by the increased area over which a high temperature is observed during the second heat-up cycle. As the surface erodes, a hole is formed. The hole increases the surface area covered by the CO₂ excitation beam, resulting in a wider temperature spread during the second heat-up cycle.

Note that although the surface was eroding and changing as a function of time, the surface temperature could be easily monitored by this fluorescence technique. Because the plastic actually burned when heated rapidly over a large area, it provided an excellent test case for studying a burning surface to determine whether the imaging system would be adversely affected by sooty flames. Initiation of burning on the surface required that a full nonapertured CO₂ beam of 80 W be incident upon the plastic surface. The resulting flame burned with a strong yellow emission characteristic of heavy soot loading. The time history of the surface temperature is shown in Fig. 11. This time history was obtained by examining only a single channel (spatial point on the surface); this represents information which would be recorded by a thermocouple or point-measurement technique. Approximately 8 s after the initiation of data acquisition, the CO₂ laser was allowed to heat the surface. The temperature rose rapidly and built to ~ 560 K. The excitation was allowed to continue for 16 s and was then extinguished. Immediately the temperature began to fall, rapidly reaching a plateau well above room temperature. The temperature of the plateau was ~ 450 K and was the result of active combustion at the surface. The surface was actively combusting due to the earlier CO₂ excitation. After ~ 7 s the flame was extinguished, and the surface temperature decayed in the expected manner.

No interference from the flame was observed nor problems caused by surface erosion. This clearly illustrates the ability of this technique to

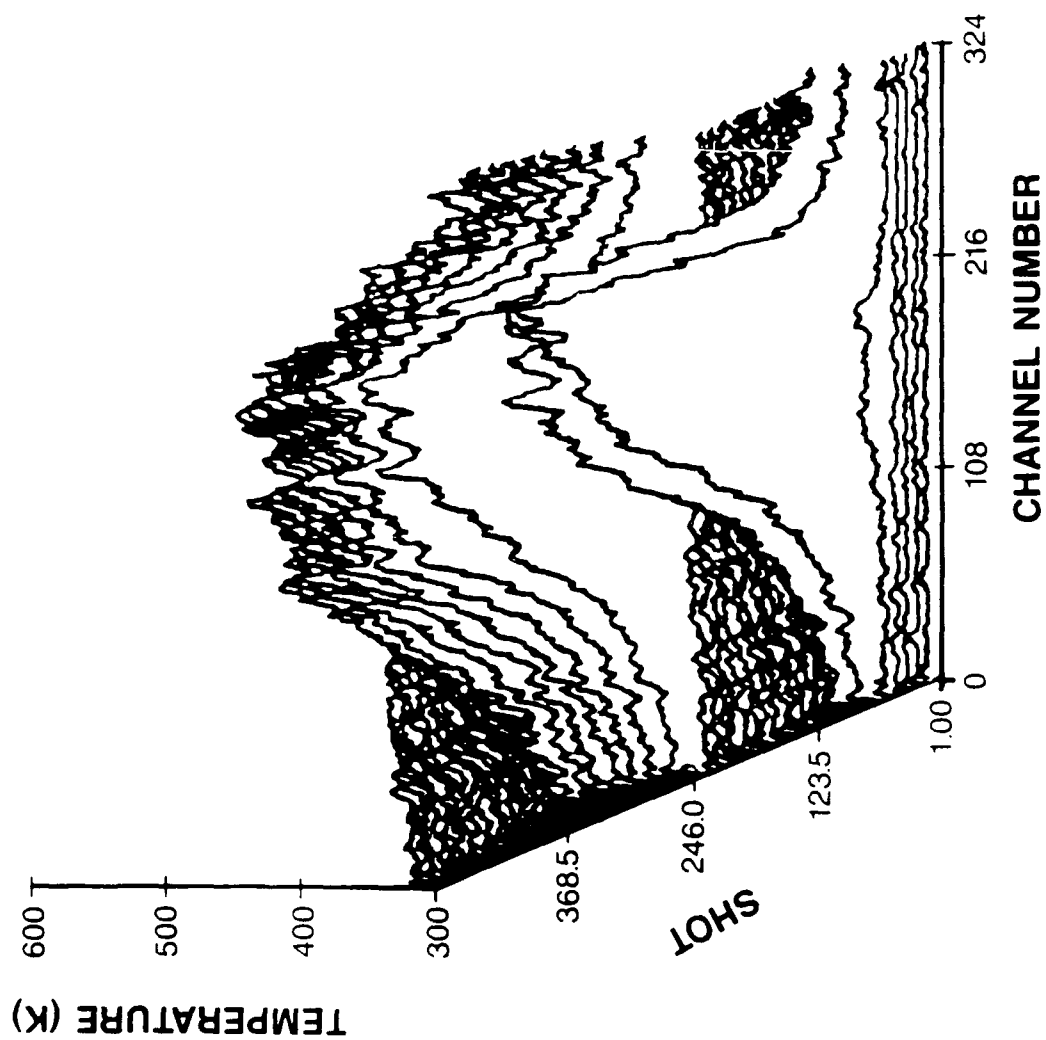


Figure 10. Plot of Temperature Resulting from CO₂ Excitation of Doped Plastic Target Undergoing Two Heating and Cooling Cycles.

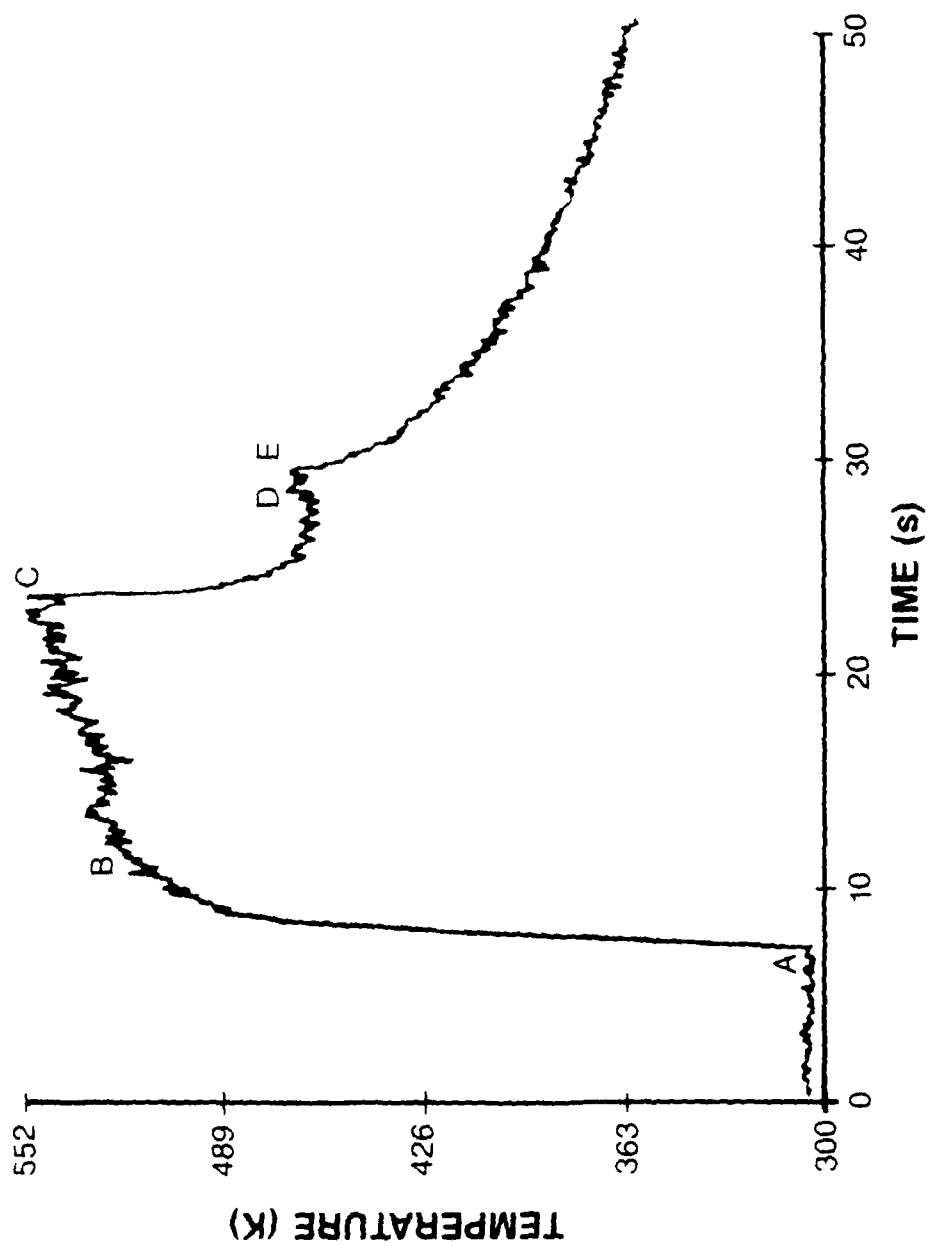


Figure 11. Plot of Temporal History of Temperature at Point on Plastic Target Doped with Dy:YAG Crystals and Heated Over Entire Surface with CO₂ Laser Beam of ~80 W. A - CO₂ Laser On; B - Target Face Burning; C - CO₂ Laser Off; D - Temperature Due to Active Combustion of Target Material; and E - Combustion Extinguished.

function effectively in adverse combustion environments on reactive surfaces.

Linear-time-resolved temperature data were also employed in the linear-depth-profile measurements discussed in the section on thermal-depth-probe studies.

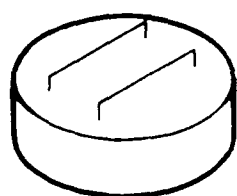
Two-dimensional imaging efforts were expended in developing the software and hardware needed to synchronize the Nd:YAG laser and the camera. Initial measurements were made on a nonreacting ceramic surface. Parallel Nichrome wires were coated with a ceramic adhesive, and the surface was doped with Dy:YAG crystals. The wires were heated electrically, and a sequence of two-dimensional images was taken as the wires heated up and cooled down. The experimental arrangement is shown in Fig. 12, and the three images from a heat-up and cool-down cycle are shown in Figs. 13-15.

Thermal-Depth-Probe Studies

Because of the importance of surface temperature and thermal penetration depth in controlling the reaction rate of energetic materials, an effort was undertaken to apply the LIF-thermographic-phosphor technique to the problem of developing a thermal depth probe. The classical approach to this measurement involves embedding small thermocouples into the sample and allowing the sample to burn through the thermocouple location. This approach requires the use of small fragile thermocouples which are destroyed during the measurement process. The thermocouples in practice must be much smaller than the desired spatial resolution in order to minimize thermal conduction along the lead wires. Problems of determining the exact location of the surface during the burn-through must also be addressed.

Linear Depth Profile

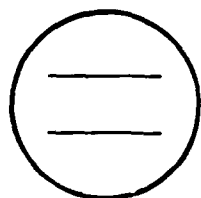
One approach in which the thermographic phosphors can be utilized in determining the thermal profile involves trimming the edge of the material to be studied to provide a flat surface and imaging the



CERAMIC BLOCK WITH
PARALLEL WIRES ABOVE
SURFACE



SIDE VIEW



TOP VIEW

Figure 12. Sketch of Ceramic Block Used for
Initial Experiment.

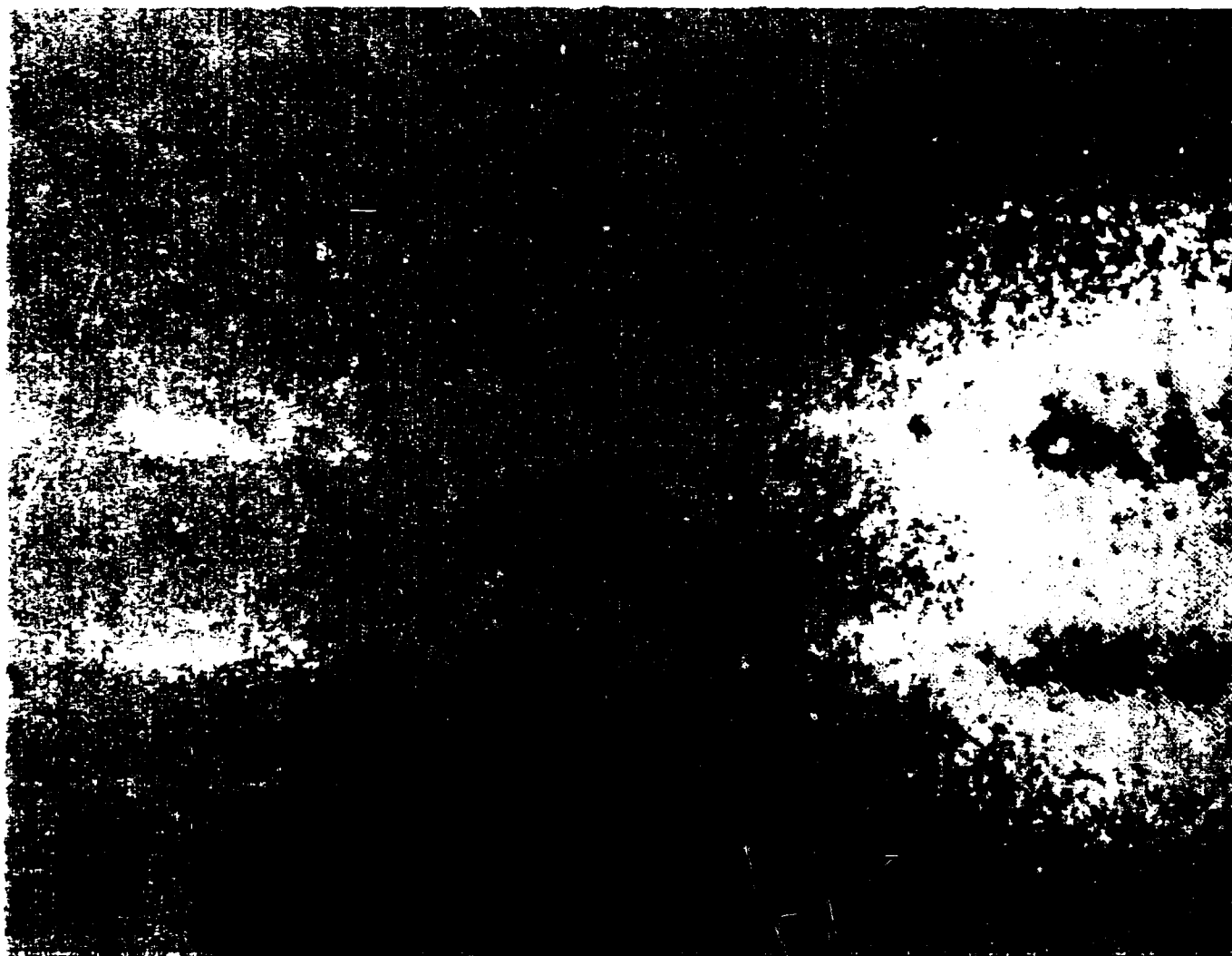


Figure 13. Increase in 467-nm Fluorescence
During Heating Cycle (2 s after
current was turned on).



Figure 14. Further Increase in 467-nm Fluorescence During Heating (8 s after current was turned on).

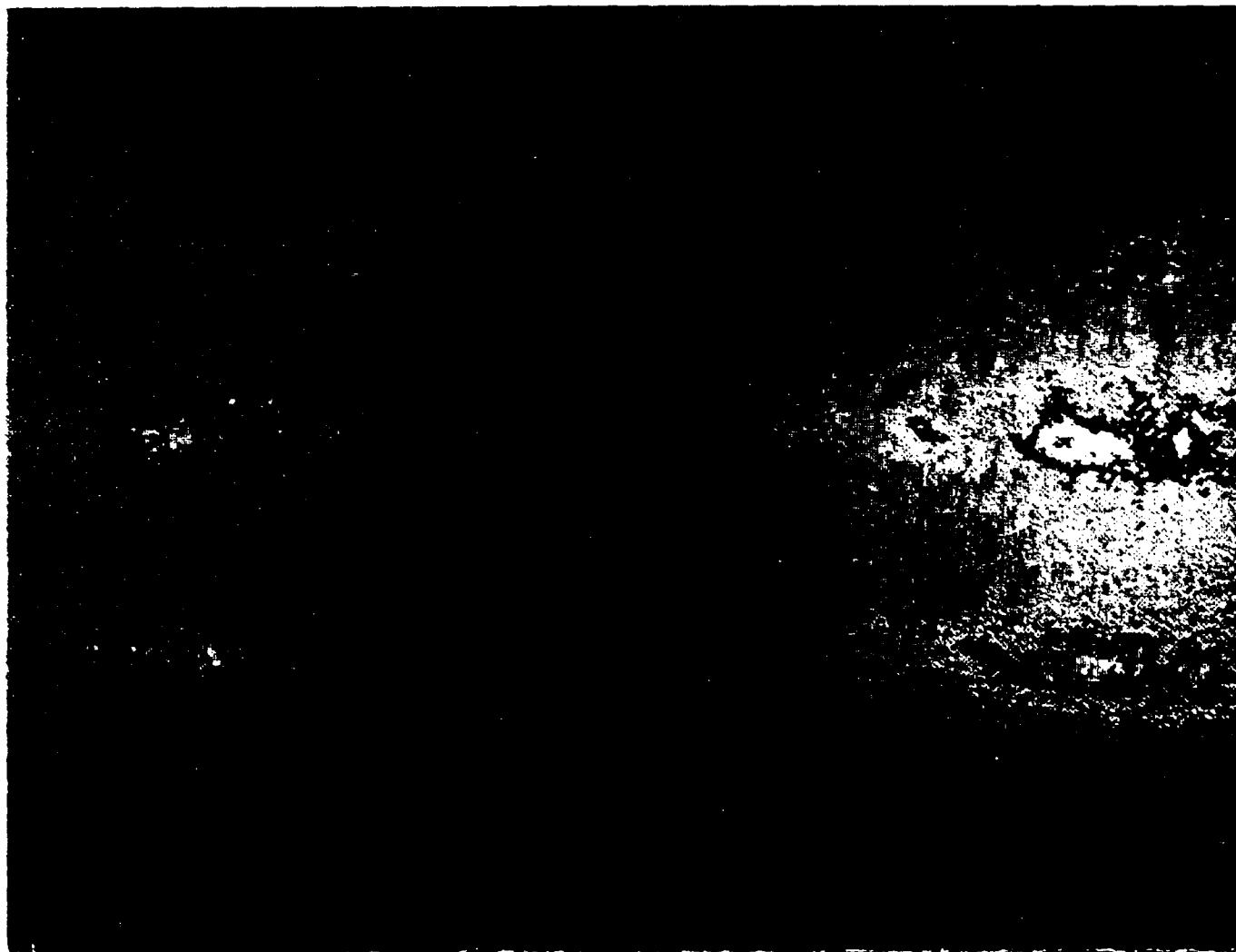


Figure 15. Decrease in 467-nm Fluorescence
When Current Discontinued (6 s
after current was turned off).

fluorescence from this side during the combustion process. This allows the entire depth profile to be monitored as a function of time--two dimensionally rather than at a single point. This approach is demonstrated in Fig. 16. In this case a CO_2 laser was used as the heat source and directed along the edge of a plastic target. The edge was illuminated by the tripled Nd:YAG beam (355 nm) and the fluorescence (temperature) recorded over a period of time. As the plastic heated and eroded, the CO_2 beam penetrated further along the edge of the sample. The temporal history of the temperature profile was monitored while this erosion was taking place. The observed thermal depth profile is displayed in Fig. 17. This figure depicts the gradual buildup of the surface temperature, the plateau at the maximum surface temperature, and the penetration of the heat into the plastic as a function of depth and time.

Fiber-Optic Probe

An alternative approach to this task was investigated by developing a fiber-optic probe which can be embedded into a sample in the same manner as a thermocouple. A fiber was used to transmit not only the exciting UV laser light but also the resulting fluorescence signal from the doped tip. The probe design is shown in Fig. 18. A beam splitter was used to separate the UV laser emission from the visible fluorescence. The fluorescent crystal was bonded to the sapphire fiber by means of a high-temperature ceramic adhesive.

The tip of the probe was inserted into an oven along with (and ~ 0.25 in. from) a Pt-Rh thermocouple. The oven was heated and maintained at a preset temperature until both the fiber-optic probe and the thermocouple yielded consistent results. The results from room temperature to 1400 K are shown in Fig. 19. Above ~ 1100 K the probe response did not increase with temperature. Subsequent examination of the probe tip indicated the possibility that a transition had occurred in the ceramic bonding material which interfered with the transmission of UV and/or visible light. Alternative methods of attaching the fluorescent crystal to the sapphire fiber must be developed in order to measure temperatures in the 1100 - 1800 K range with this technique.

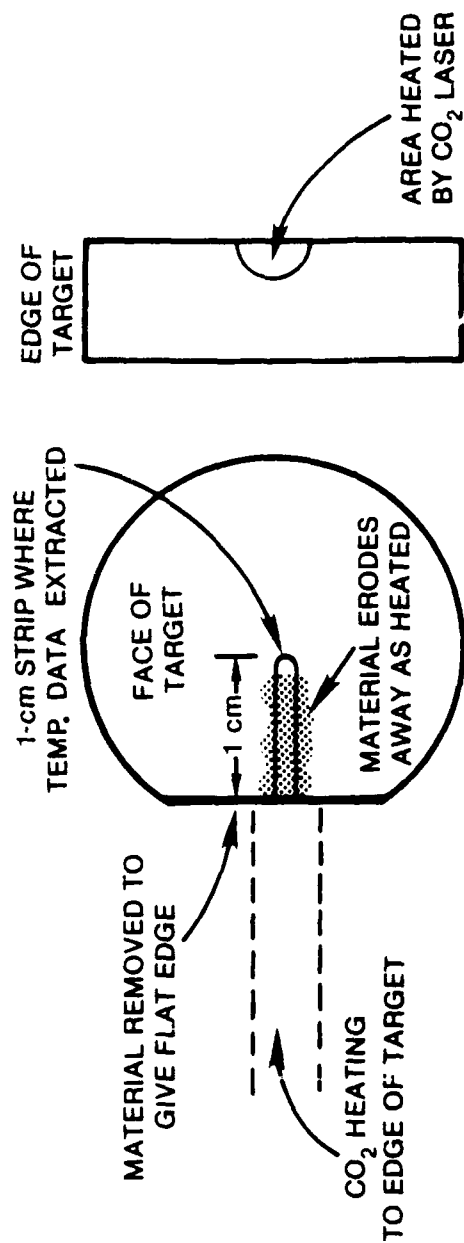


Figure 16. Schematic Diagram of Method for Obtaining Thermal Depth Profile of Energetic Material.

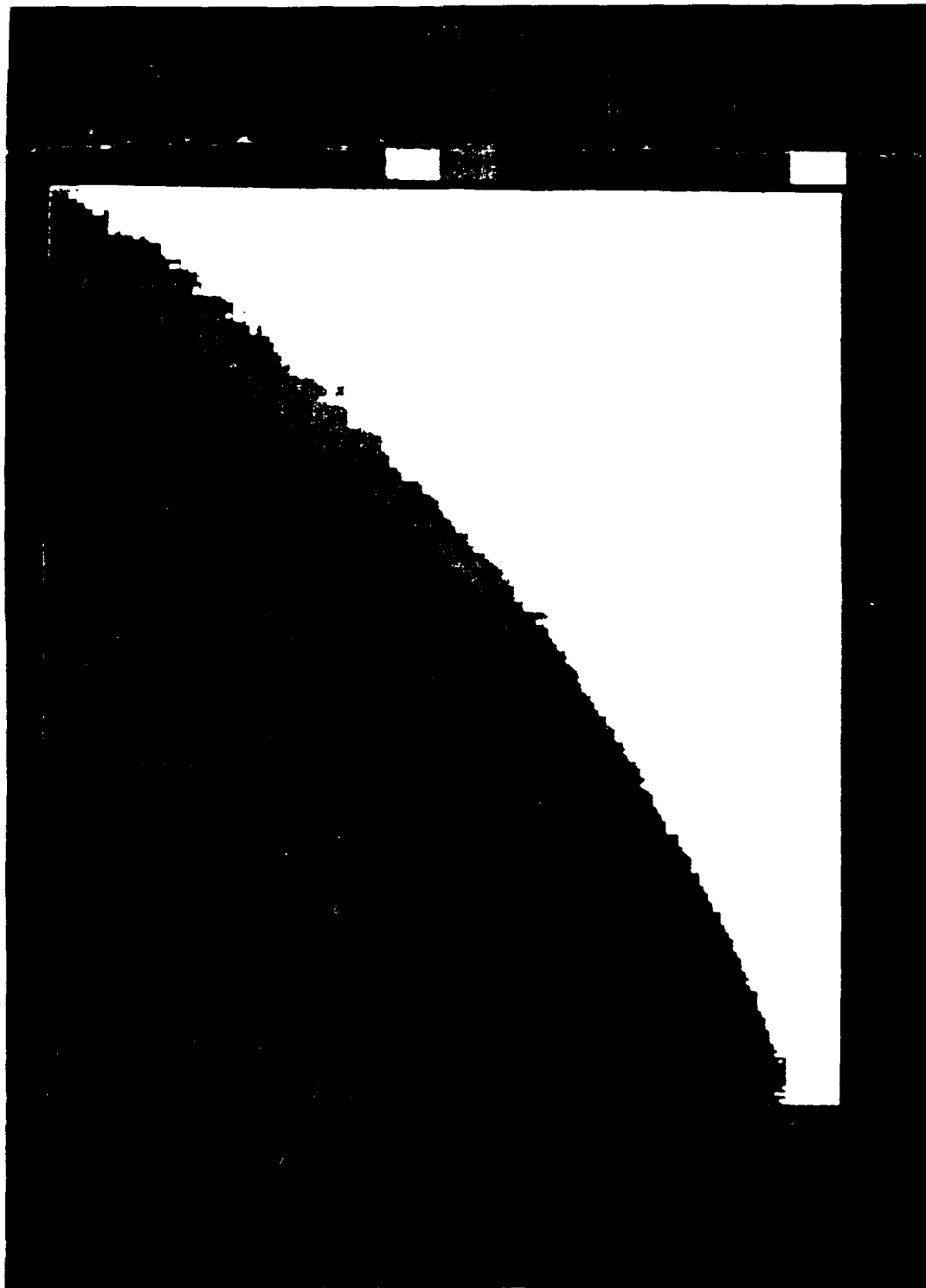


Figure 17. Color Contour Displaying Thermal Depth Profile of Plastic Target Heated with cw CO_2 Laser ~ 30 W.

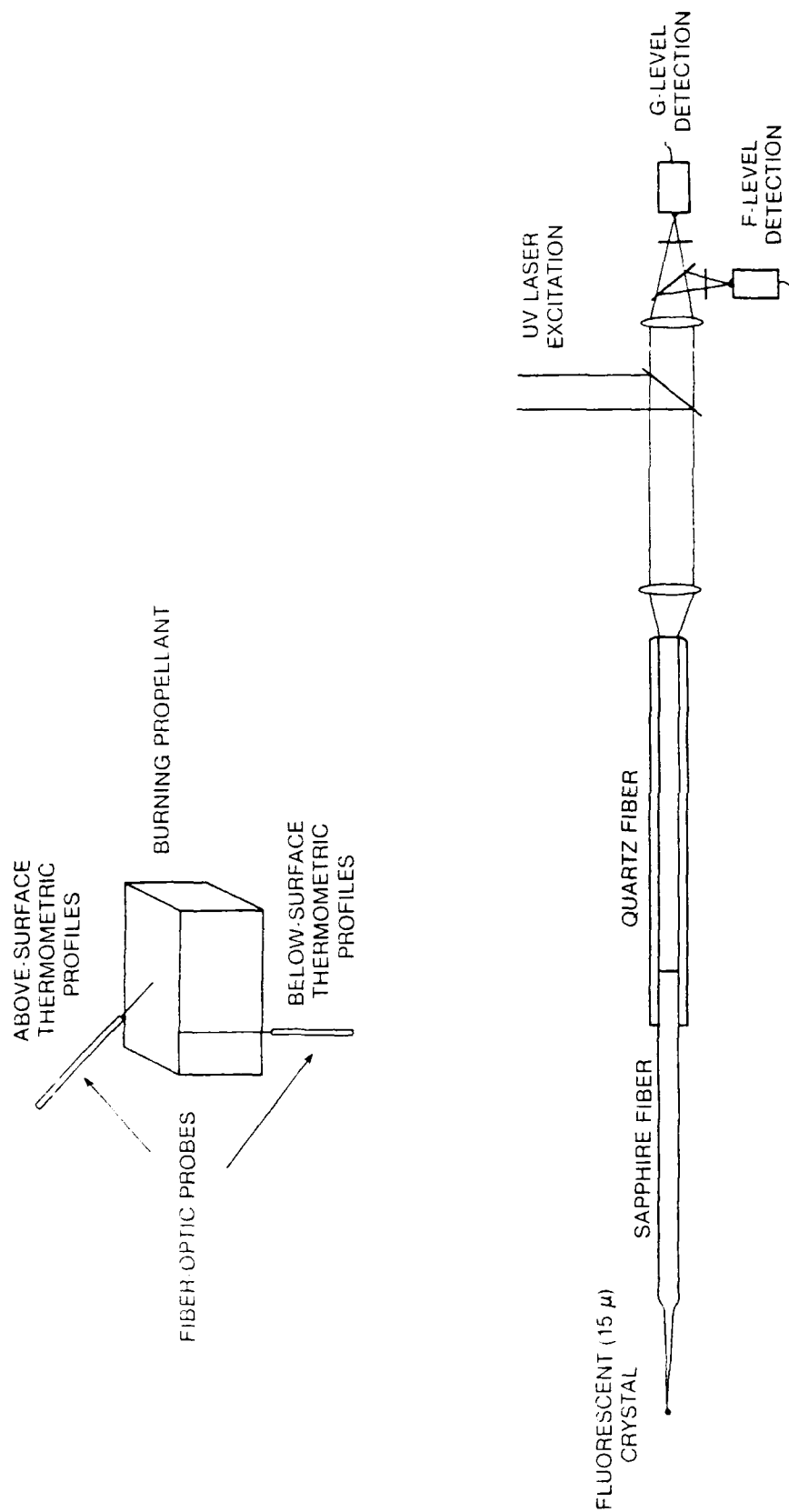


Figure 18. Fiber-Optic Probe for Thermal Depth Profiling.

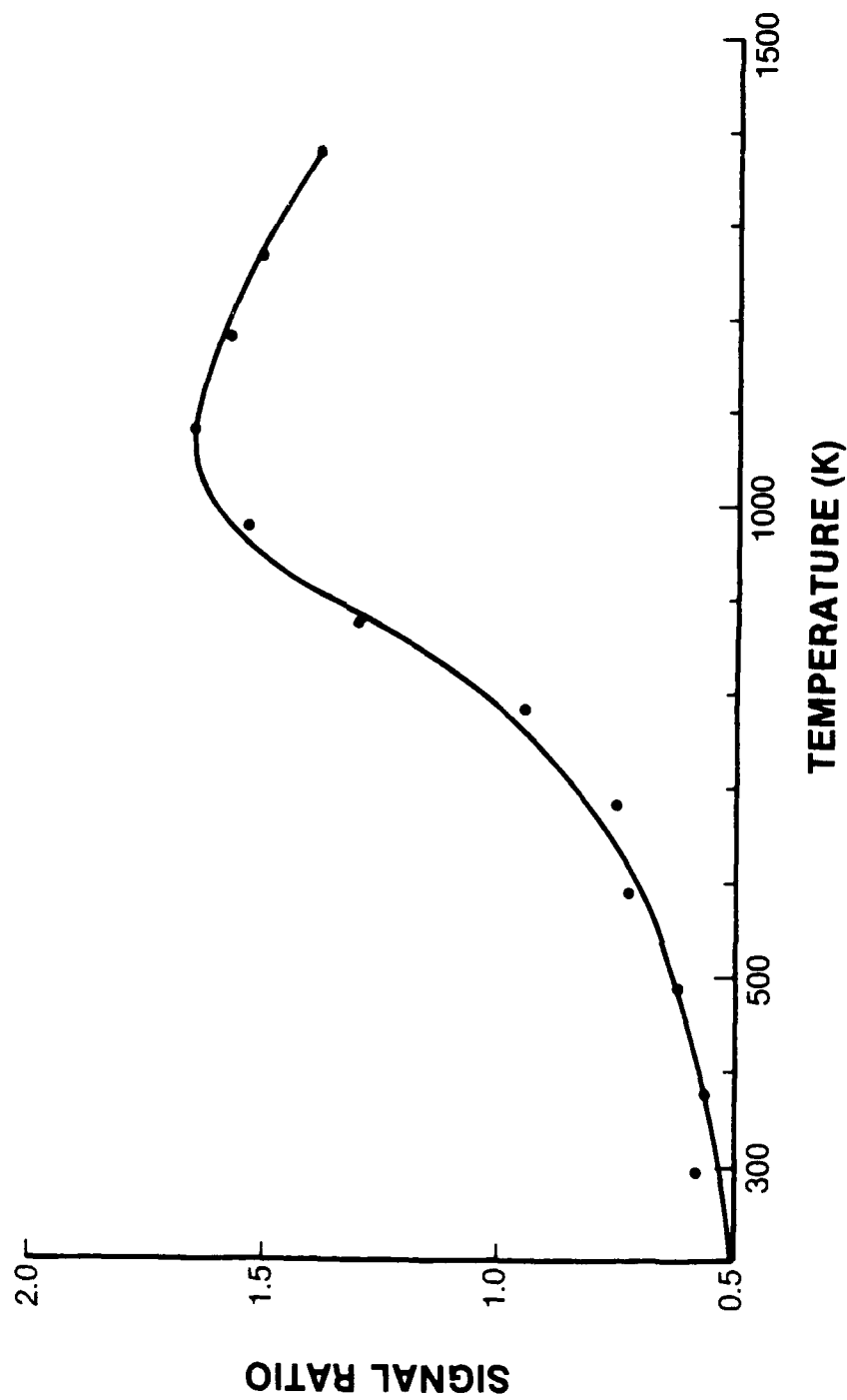


Figure 19. Plot of Signal Response as Function of Temperature for Fiber-Optic Probe.

The advantage of the probe configuration is that it is single-ended and can be used in limited-optical-access situations. In addition, the optical signal is not subject to some of the interferences that can affect the electrical signals of thermocouples. The optical fiber is also inert to most chemical environments.

The disadvantages of the probe configuration are essentially the same as those of thermocouples. Good thermal contact with the surrounding sample must be considered. Temporal response is dependent upon the thermal properties and size of the fluorescent crystal, as in the case of a thermocouple tip. Conduction heat transfer down the probe can also affect the temperature measurement.

Nonmetallic Materials and Surface-Bonding Studies

In order to apply point, line, or surface LIF temperature-measurement techniques to other materials or surfaces, methods for bonding the Dy:YAG crystals to a variety of surfaces must be considered. Techniques that result in a relatively thin layer of crystals which are in good thermal contact with the surface are of primary interest. Techniques such as sputtering, electron-beam deposition, flame spray, and plasma spray have been considered. Dy:YAG crystals can be melted and recrystallized without altering the properties which make them desirable for LIF. Sputtering and electron-beam deposition methods are generally used for metals, and most of the readily available equipment which makes use of these methods can accommodate only small objects ($2 - 5 \text{ cm}^2$). Flame-spray is more portable and applicable to objects of diverse geometries, but it has a lower impact velocity than plasma spray. Plasma spraying allows a strong bond to be formed, and bonding to ceramic materials is especially effective.

In addition to thermal bonding techniques, methods have also been considered which may employ high-temperature adhesives. These materials include silicates, ceramics, and epoxies. Two-dimensional imaging of heated, inert material was demonstrated by bonding Dy:YAG crystals to the surface of a high-temperature ceramic adhesive. In these studies this method was found to be the most convenient and useful for bonding crystals to surfaces of interest.

Section III

PRESENTATIONS AND PUBLICATIONS

The following presentations and publications have resulted from the research performed on this program:

"Surface Thermometry of Energetic Materials by Laser-Induced Fluorescence," L. P. Goss and M. E. Post, Presented at the AFOSR/ONR Propulsion Contractors Meeting, 13-17 June 1988, Monrovia, California.

"Surface Thermometry of Energetic Materials by Laser-Induced Fluorescence," M. E. Post and L. P. Goss, Presented at the AFOSR/ONR Propulsion Contractors Meeting, 19-23 June 1989, Ann Arbor, Michigan.

"Surface Thermometry by Laser-Induced Fluorescence," L. P. Goss, A. A. Smith, and M. E. Post, Accepted in August 1989 for Publication in the Review of Scientific Instruments.

"Thermal Depth Measurements Using Laser-Induced Fluorescence," L. P. Goss and M. E. Post (In Preparation), To be Submitted to Optics Letters.

Section IV

PROFESSIONAL PERSONNEL ASSOCIATED WITH RESEARCH EFFORT

The following professional personnel have been associated with the research effort on this contract:

Michael E. Post
Research Physicist

Larry P. Goss, Ph.D.
Principal Investigator

Section V

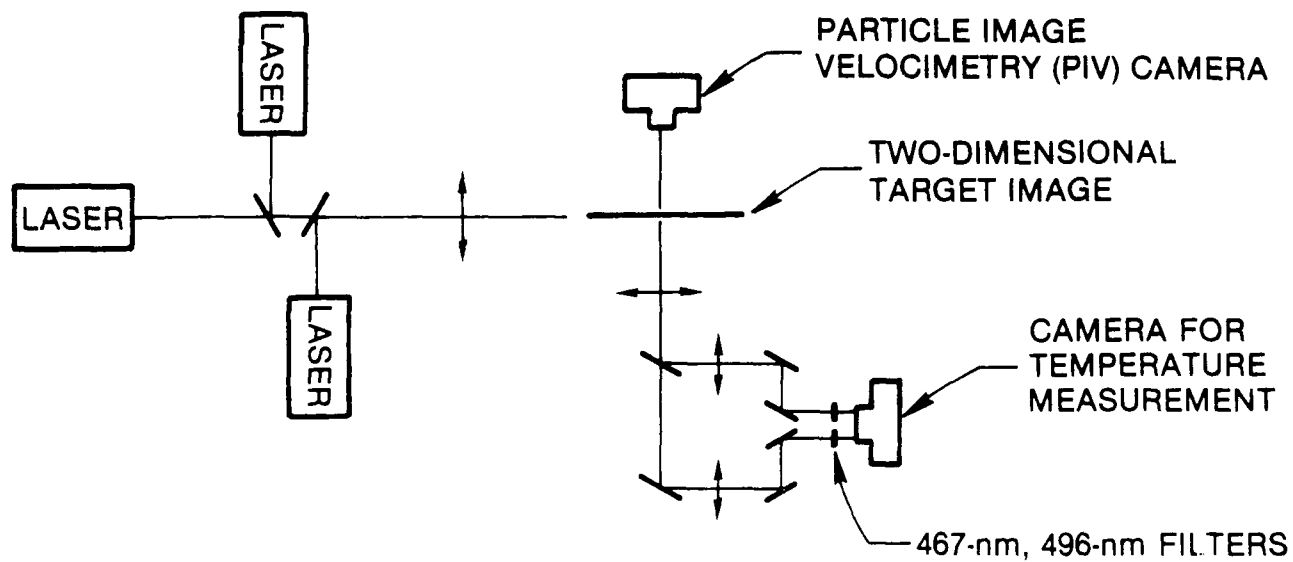
RECOMMENDATIONS FOR FUTURE STUDIES

The type of research described herein inevitably leads to identification of areas for additional refinement or new developments. Some of these areas for future study are discussed below.

Simultaneous Temperature and Velocity Measurements in Gas Flows

Results of two-dimensional temperature measurements on a variety of surfaces such as inner walls of operating engines, rotating surfaces in engines, and surfaces of energetic materials would be of great interest. This technique could be extended to flame surfaces or hot gases by seeding with micron-size Dy:YAG particles. It may be possible to combine particle-image-velocimetry (PIV) techniques and LIF techniques using these seed particles to obtain near-simultaneous temperature and velocity information over a two-dimensional image. An experimental design for making this type of measurement is shown in Fig. 20.

Two-dimensional velocity flowfield measurements could be made using two-color PIV with Dy:YAG seed particles. Two-color PIV is similar to existing PIV techniques except that two different-color laser sources are used to form the light sheets required for exposing the position of particles in a seeded flowfield. A green-colored laser sheet (formed by a doubled Nd:YAG laser) and a red-colored laser sheet (formed by a Nd:YAG-pumped dye laser) are employed sequentially to expose the particle positions which are recorded on 35-mm color film. Analysis of the resulting images involves digitizing the exposed film with color filters to separate the green- and red-particle image fields and processing the digitized images with velocity-displacement software. The two-color PIV technique has the advantage that direction, as well as particle displacement, is uniquely determined because the green-particle image occurs before the red one by a known time increment.



2 LASERS PROVIDE COLOR-CODED PIV PULSES

1 LASER PROVIDES LIF EXCITATION PULSE

Figure 20. Proposed Arrangement for Simultaneous Temperature and Velocity Measurement for Gas Flows.

In complex flowfields (i.e., recirculating flows), it is important to obtain an instantaneous two-dimensional picture of the entire flowfield rather than a time-averaged pointwise map. The two-color PIV technique is designed to remove directional ambiguities, allowing measurements to be made in complex flow systems. The technique consists of employing small particles to seed a gas stream exiting a nozzle or passing through a test section. A laser sheet (green), formed by passing the output from a doubled Nd:YAG laser through a cylindrical lens, is used to illuminate the seed particles in the flowfield. After a short selected time interval (ranging from a few microseconds for high-velocity flow systems to milliseconds for low-velocity flow systems), a second laser sheet (red) from a Nd:YAG-pumped dye laser is pulsed to illuminate the flowfield a second time. Any particles remaining in the plane of the laser sheets produce a "displacement-vector" pair comprised of a green- and a red-particle color film and processed by direct digitization. The unique color coding helps to remove directional ambiguities associated with single-color techniques because the red image occurs after the green one.

The two-dimensional temperature image would be taken by exciting the Dy:YAG seed particles with the tripled output of a Nd:YAG laser at some time between the laser pulses for the two-color PIV.

Surface-Temperature Measurements for Energetic Materials

One of the significant accomplishments of this project was measurement of surface temperature on a burning plastic target. Dy:YAG crystals were mixed into the plastic, and temperature measurements were made while the UV-excitation pulses and optical signal traveled through a sooting flame. Additional work with energetic materials appears to be an area which would be of significant fundamental and practical importance. Solid propellants are widely used, but information concerning the temperature of the reacting surface is extremely limited.

Further Development of Fiber-Optic Probe

Additional work on the tip configuration and bonding methods could produce a fiber-optic probe for point measurements in the 300 - 1800 K temperature range.

Surface-Bonding Studies

The most convenient method for bonding Dy:YAG crystals to surfaces of interest proved to be the use of high-temperature adhesives. Additional studies on the performance of these adhesives over a range of duty cycles would be useful. Plasma spraying and flame spraying were identified as prime candidates for applying Dy:YAG crystals directly onto a surface. Studies which consider surface smoothness and the effect of various temperature duty cycles would be useful.

Section VI

SUMMARY

The most significant accomplishment on this program was the demonstration of the two-dimensional thermal imaging of heated inert surfaces. Thermal depth profiling has been conducted with a linear array, and a fiber-optic thermal probe has been developed for the 300 - 1100 K range. In efforts on bonding techniques of Dy:YAG crystals, plasma spray was found to hold the most promise for nonmetallics. Ceramic adhesives were used in most surface applications for this program.

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2. L. P. Goss and M. E. Post, "Surface Thermometry of Energetic Materials by Laser-Induced Fluorescence," Annual Technical Report on AFOSR Contract F49620-87-C-0040, covering the period 17 February 1987 - 17 February 1988 (Systems Research Laboratories, Inc., Dayton, OH, April 1988).

Appendix A

SURFACE THERMOMETRY BY LASER-INDUCED FLUORESCENCE

Surface Thermometry by Laser-Induced Fluorescence

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ABSTRACT

A novel laser-induced fluorescence technique has been developed for measuring the surface temperature of reacting and non-reacting materials. The technique involves seeding the material to be examined with a temperature-sensitive phosphor (Dy:YAG) and monitoring the laser-induced fluorescence of the phosphor to determine the temperature. The Dy:YAG phosphor displays a temperature sensitivity in the range 300 - 1700 K. The technique has been applied to both reacting and nonreacting surfaces under laser excitation, allowing temperature and temporal-history profiles to be determined.

INTRODUCTION

The need for a probe for examining surface thermometry of reacting and nonreacting materials is evident, for example, in the study of condensed-phase combustion processes of energetic materials. Such studies in intermediate- (3 - 20 MPa) and high- (> 300 MPa) pressure regions present a number of challenges for researchers in the field of optical diagnostics; to date, this area has received less attention than the experimentally more-tractable problem of gas-phase combustion. The choice of a diagnostic technique for surface-temperature measurements is complicated by the requirements of high spatial resolution (1 μm), high temporal resolution (> 1 μs), nonintrusiveness, remote probing, and making measurements on an ever-changing reacting surface.

Advanced optical diagnostics such as coherent anti-Stokes Raman spectroscopy (CARS),¹ ordinary Raman spectroscopy,² and laser-induced fluorescence (LIF)³ have been employed extensively for combustion measurements in atmospheric flames and plumes of energetic materials. However, no attempt has been made to apply these techniques to the measurement of reacting-surface temperatures. Of the available optical

diagnostic techniques, LIF was considered to hold the most promise of meeting the criteria for an effective diagnostic method for surface-temperature measurements. However, in propellants or other materials of interest, no molecular or atomic species exists naturally which can be used for LIF surface-temperature measurements. Thus, for this technique to be employed, it was necessary to introduce into the material of interest a fluorescent species whose emission spectrum displayed sensitivity to temperature over the temperature range of interest. The rare-earth ion dysprosium (Dy) doped into a yttrium-aluminum-garnet (YAG) crystal was determined to be an ideal fluorescent species for thermometry.⁴⁻⁵

The LIF technique described in this paper involves embedding small-sized (10 - 100 μm) crystals of a temperature-sensitive phosphor ($\text{Dy}^{+2}:\text{YAG}$) in the surface of the material whose temperature is to be measured. Fluorescence from the Dy:YAG crystals, induced by the tripled output of a Nd:YAG laser source, is collected and used to determine the surface temperature.

THEORY

Temperature may affect the spectrum of rare-earth ions in several ways.⁶ First, the addition of heat causes the crystal lattice containing the rare earth to vibrate which, in turn, creates a changing crystal field at the location of the rare-earth ion and produces a broadening in the linewidth of the optical transitions. Secondly, as the temperature is increased, the number of upper levels of the system which become populated increases according to Boltzmann's law, $\exp(-e/kT)$. This "thermalization" effect causes a change in the intensity distribution of the rare-earth spectrum with temperature. Thirdly, a frequency shift of the spectral lines can occur due to the thermal expansion of the crystal lattice. The greater average ion separation at high temperature results in a reduced crystal field and, in turn, reduced Stark-component separation. Finally, as the temperature increases, the vibration of the crystal lattice (phonons) nonradiatively relaxes the excited electronic state, thus reducing the lifetime of the radiative transition (quenching). Of these effects, the "thermalization" process was determined to be the optimum technique for surface thermometry.

The "thermalization" process of these rare-earth compounds can be explained by the close spacing of their energy levels. When two rare-earth energy levels are separated by $\sim 1000 \text{ cm}^{-1}$, the upper level typically will not fluoresce at low temperature due to extremely high multi-phonon relaxation rates which act to quench these closely spaced levels. Thus, at low temperatures, no population buildup occurs in the upper energy level and, therefore, no fluorescence is observed. As the temperature increases, the upper level becomes more populated and the fluorescence from this level increases. Thus, by monitoring the increase in fluorescence of the upper level relative to the lower, the temperature can be determined.

This type of behavior is exemplified in trivalent dysprosium, doped at 3% in yttrium-aluminum-garnet ($\text{Dy}^{+3}:\text{YAG}$) (simplified energy diagram shown in Fig. 1). Absorbed laser light excites Dy^{+3} into a high-energy state which radiatively and nonradiatively decays to the F-level. This level undergoes a fast thermal equilibrium which pumps a portion of its population into the nearby G-level. The fluorescence is then observed from both states. Figure 2 displays the fluorescence from both the F- and G-levels of $\text{Dy}:\text{YAG}$, including various Stark-shifted components, as a

function of temperature. The relative intensity of the F-fluorescent level located at 496 nm remains fixed as the temperature increases, while the intensity of the G-level component located at 467 nm increases dramatically with temperature.

It is evident from Fig. 2 that several regions of the Dy:YAG spectrum are temperature sensitive and, therefore, useful for thermometry. It is also evident that the 496-nm peak can be used for an internal standard, allowing the temperature determination to be a relative measurement rather than a more-difficult absolute one. The ratios of the peaks which are blue in wavelength display behavior similar to that of the 467-nm peak which displays high-temperature sensitivity up to 1800 K. Above this value the fluorescence intensity drops below detectable limits. The 512-nm peak ratio is different in that it is insensitive to temperatures below 500 K but then rapidly increases in sensitivity at high temperatures.

The fluorescence from the G- and F-levels of Dy:YAG extinguishes at a temperature of ~ 1800 K due to the large phonon quenching rates at this high temperature; the melting temperature of the YAG crystal is ~ 2100 K.

This operating range is more than adequate for monitoring surface temperatures on most reactive materials.

EXPERIMENTAL

One of the major advantages of employing LIF of thermographic phosphors is the possibility of using one- and two-dimensional detectors to record line and entire-surface temperature distributions. By simultaneous monitoring of the F- and G-levels of Dy:YAG, the surface temperature at a point along a line or over the entire surface can be determined with appropriate detectors.

The experimental arrangement used for surface thermographic imaging in this study is shown in Fig. 3. The main detector component of the system is a 1024-element intensified diode array (Tracor Northern TN1710). The fluorescence from the surface is excited by the 355-nm tripled output of a Quanta-Ray DCR-1A Nd:YAG laser. The output of the laser is an ~ 8 ns pulse which pumps the Dy:YAG into a highly excited electronic state. The intensified detector is gated with a 10- μ s pulse which is an aid in discriminating against unwanted background radiation from the surface. This

feature is very important at elevated temperatures where the natural blackbody emission from the surface and surroundings can be particularly high.

The fluorescence from the surface is collimated by an F-6 lens, split into two equal paths, and directed through 496- and 467-nm filters for the F- and G-level fluorescence, respectively; the two paths are imaged side by side on the DARSS detector. Great care was taken to prevent path differences between the two legs; matched imaging lenses were used, which helped to ensure that the images at the two wavelengths would be spatially identical.

The video signal from the detector is digitized, stored in a temporary memory, and subsequently sent to a minicomputer. The data are stored on disk for analysis at a later date. The limited rate of data transfer is determined by the 10-Hz operation of the Nd:YAG laser. Image analysis consists of ratioing the F- and G-fluorescence signals and converting the resulting signal to a temperature with the aid of calibration curves established using a platinum-coiled oven. The surface temperature can then be examined as a function of time and/or space.

Calibration of the system was performed with a platinum-coiled oven which is capable of temperatures in excess of 1723 K. The calibration target was ceramic formed around a Pt-Rh thermocouple. The face of the ceramic was coated with a layer of Dy:YAG particles, $< 20 \mu\text{m}$ in diam. The overall oven temperature was monitored with another Pt-Rh thermocouple mounted ~ 1.25 cm above the target. As each preset temperature was achieved, the oven was held at that temperature for 30 min. to ensure overall temperature stability. The first setting was at room temperature (~ 293 K) and the second at 323 K. Subsequent temperature settings were in 50-K increments to a maximum of 1673 K. A plot of the ratio of the 467- to the 496-nm intensity is shown in Fig. 4.

The LIF signal is very reproducible as a function of temperature; thus, the accuracy of the LIF technique directly corresponds to that of the thermocouple used for calibration which is in the range 1 - 10 K, depending upon the absolute temperature. The precision of the measurement is affected by a number of factors, including photon statistics, detector noise, and background signal levels. With the large signal levels from the Dy:YAG crystals, the major limit to precision is the detector noise which is $\sim 3\%$.

Thus, in a single-shot mode of operation, a $\pm 9 - \pm 50$ K uncertainty is observed over the 300 - 1500 K temperature range.

RESULTS AND DISCUSSION

Measurements on a nonreacting surface were made using the experimental arrangement shown in Fig. 5(a). The surface consisted of small ($< 50 \mu\text{m}$) Dy:YAG crystals implanted in a ceramic material in which a small Nichrome wire had been embedded. An electrical current was allowed to flow through the wire to act as a local heat source, and the fluorescence from the 467-nm G-Stark component was monitored by a linear multi-channel detector along a line perpendicular to the wire filament. The fluorescence was recorded in a single 10-ns pulse and stored by the computer to obtain a time history of the temperature changes at the surface under heat-up and cool-down conditions [5(b-c)]. Under heat-up conditions [5(b)], the initial rise in temperature was centered at the embedded wire, with a gradual transfer of heat to the surrounding surface. After 22 sec, the maximum temperature rise was observed in the area surrounding the embedded wire (400 K). The heat buildup beyond this point was slower, asymptotically approaching 420 K. The cooling of the surface after

interruption of electrical current to the wire filament is shown in Fig. 5(c). The temperature decline is steep in the peak-temperature area but slows as the central temperature approaches that of the surrounding heated area.

To demonstrate the thermometric technique on a surface similar to that of an energetic material, a thermal-setting plastic supplied by Buehler, Ltd., was employed. The Dy:YAG crystals were mixed in a one-to-seven weight ratio with the plastic which was heated under pressure to form the test specimen. The plastic surface erodes as it is heated above 400 K, thus, the interdispersed crystals are continuously replenished at the surface. Therefore, no loss in the fluorescence signal is observed as the surface erodes; and the surface-temperature profile can be obtained, even on reactive surfaces.

A 2-mm-focal-spot 14-W CO_2 beam was used to heat the plastic surface. The area imaged by the DARSS detector corresponds to 1 cm, which translates into $\sim 27.5 \mu\text{m}$ channel. As in the case of the ceramic specimen, the CO_2 beam is incident upon the sample at an angle of 45 deg. The excitation of the plastic surface was recorded over two heat-up and cool-down

cycles; the results are depicted in Fig. 6. Under excitation by the CO₂ laser, the plastic surface is actually eroded, as evidenced in Fig. 6 by the increased area over which a high temperature is observed during the second heat-up cycle. As the surface erodes, a hole is formed. The hole increases the surface area covered by the CO₂ excitation beam, resulting in a wider temperature spread during the second heat-up cycle.

Note that although the surface was eroding and changing as a function of time, the surface temperature could be easily monitored by this fluorescence technique. Because the plastic actually burned when heated rapidly over a large area, it provided an excellent test case for studying a burning surface to determine whether the imaging system would be adversely affected by sooty flames. Initiation of burning on the surface required that a full non-apertured CO₂ beam of 80 W be incident upon the plastic surface. The resulting flame burned with a strong yellow emission characteristic of heavy soot loading. The time history of the surface temperature is shown in Fig. 7. This time history was obtained by examining only a single channel (spatial point on the surface), which represents information which would be recorded by a thermocouple or point-measurement technique. Approximately 8 sec. after the initiation of data

acquisition, the CO_2 laser was allowed to heat the surface. The temperature rose rapidly and built to ~ 560 K. The excitation was allowed to continue for 16 sec. and was then extinguished. Immediately the temperature began to fall, rapidly reaching a plateau well above room temperature. The temperature of the plateau was ~ 450 K and was the result of active combustion at the surface. The surface was actively combusting due to the earlier CO_2 excitation. After ~ 7 sec. the flame was extinguished, and the surface temperature decayed in the expected manner.

No interference from the flame was observed nor problems caused by surface erosion. This clearly illustrates the ability of this technique to function effectively in adverse combustion environments on reactive surfaces.

ACKNOWLEDGEMENTS

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FIGURE CAPTIONS

Figure

- 1 Simplified energy diagram of F- and G-levels of Dy:YAG.
- 2 Plot of fluorescence signals of F- and G-levels of Dy:YAG as function of temperature.
- 3 Schematic diagram of system for LIF probe for surface thermometry of reacting and nonreacting materials.
- 4 Plot of ratio of intensity of G-level (467-nm) Stark component to F-level (496-nm) Dy:YAG as function of temperature.
- 5 (a) Experimental setup for surface-thermometry studies; (b). (c) Temporal variation of surface temperature of ceramic material under heat-up and cool-down conditions, respectively.

- 6 Plot of temperature resulting from CO₂ laser excitation of doped plastic target undergoing two heating and cooling cycles.
- 7 Plot of temporal history of temperature at point on plastic target doped with Dy:YAG crystals and heated over entire surface with CO₂ laser beam of ~ 80 W. A - CO₂ laser on; B - target face burning; C - CO₂ laser off; D - temperature due to active combustion of target material; and E - combustion extinguished.

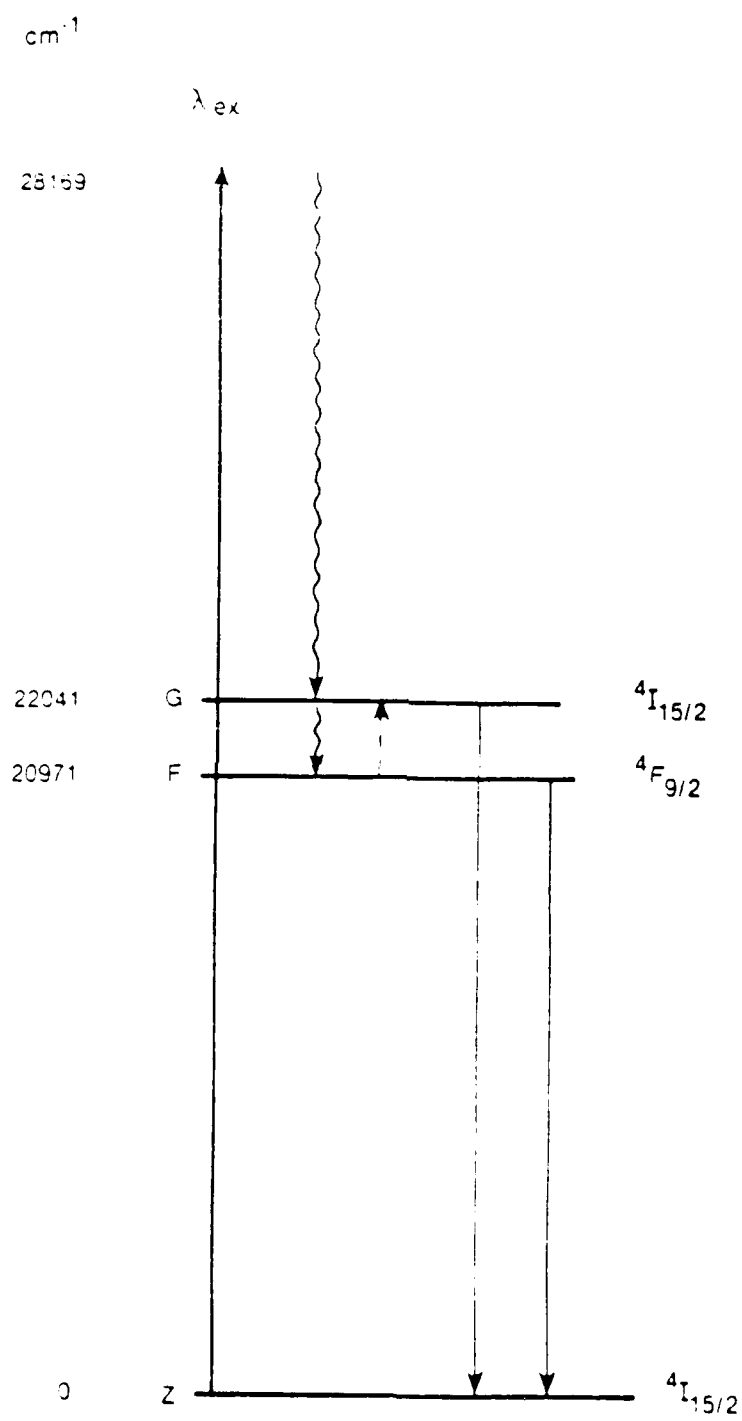


Figure 1

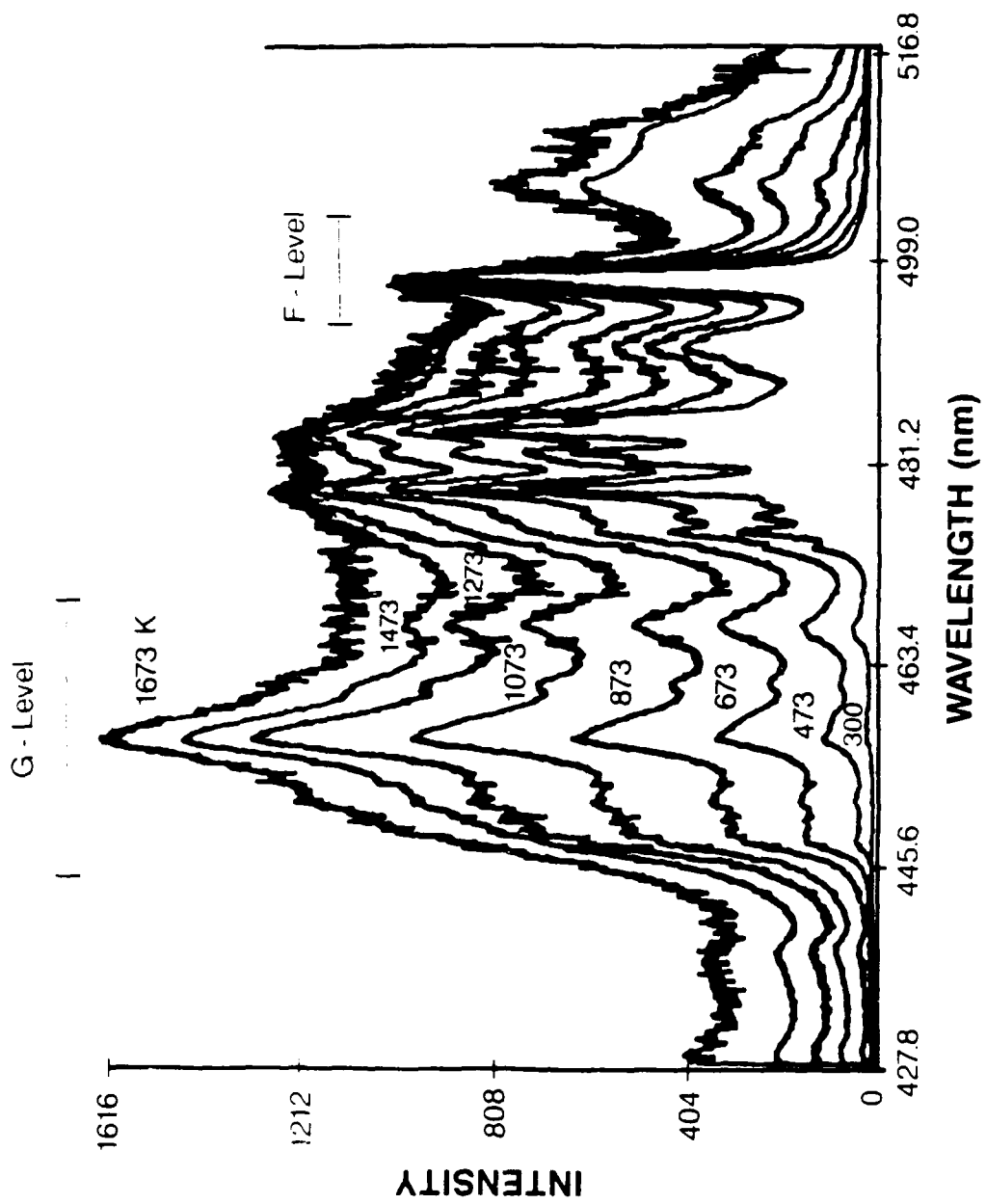


Figure 2

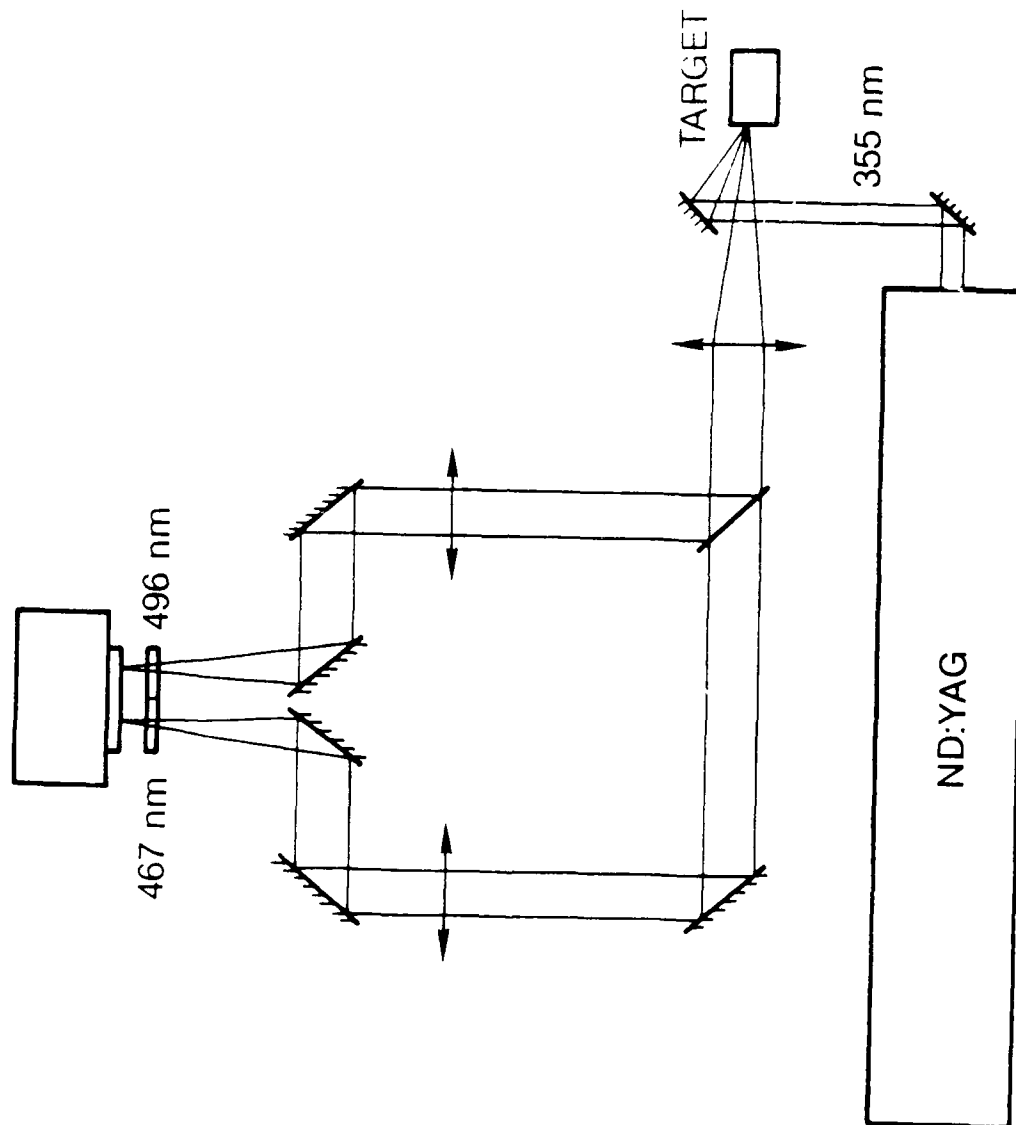


Figure 3

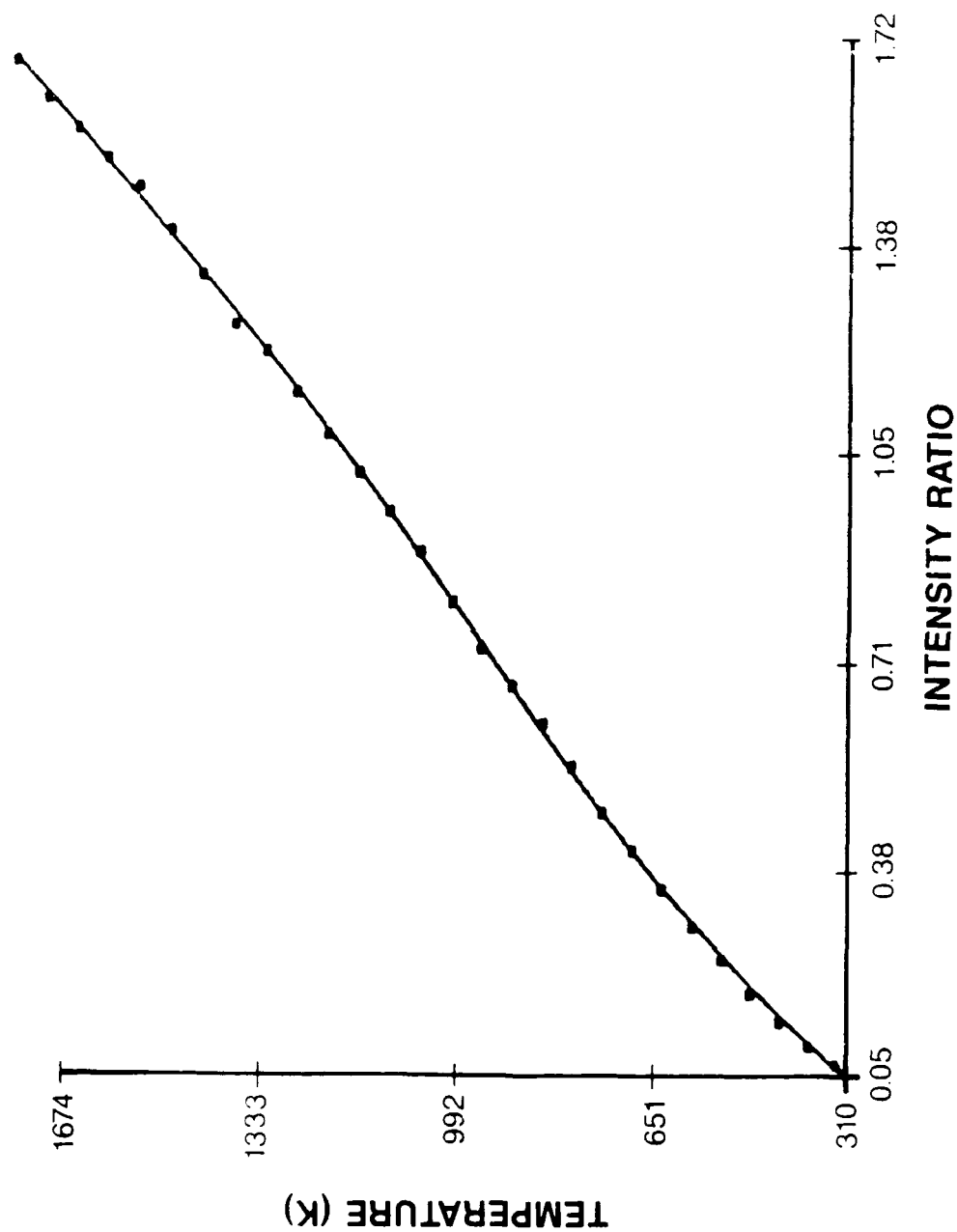


Figure 4

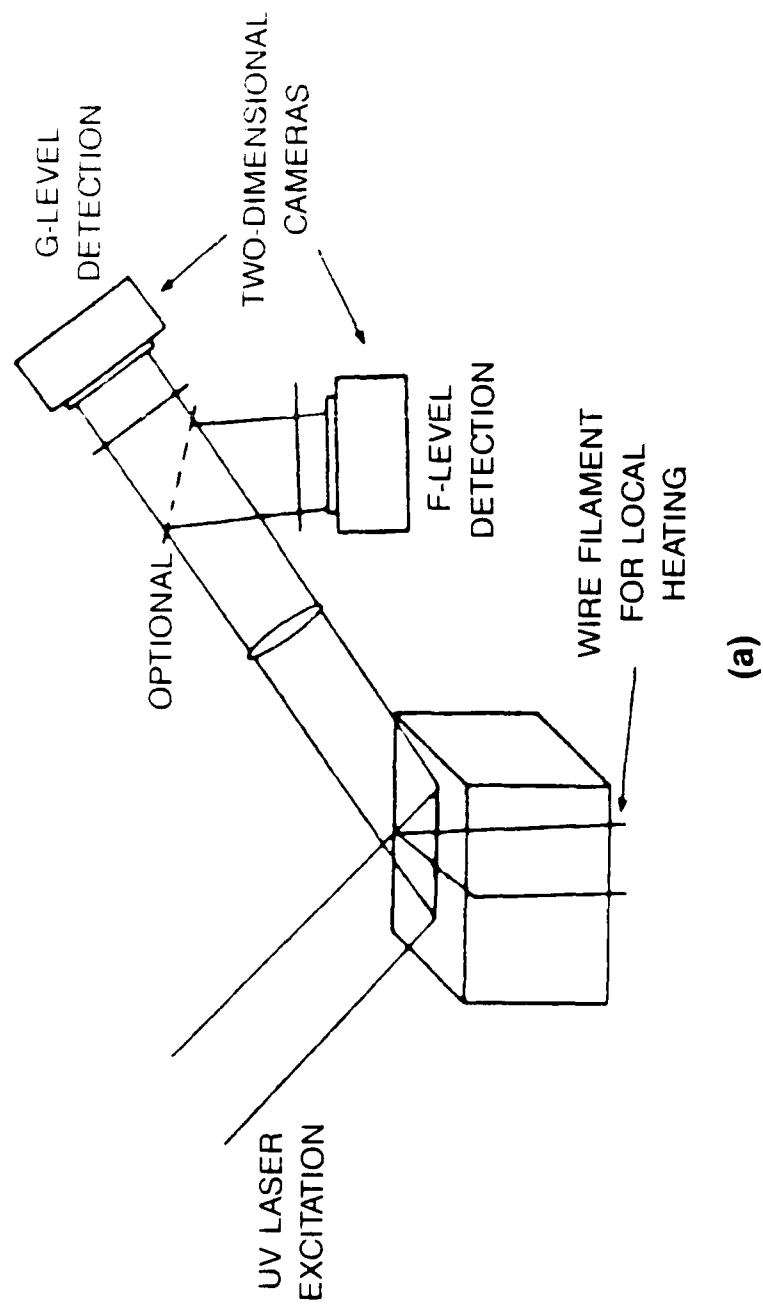


Figure 5(a)

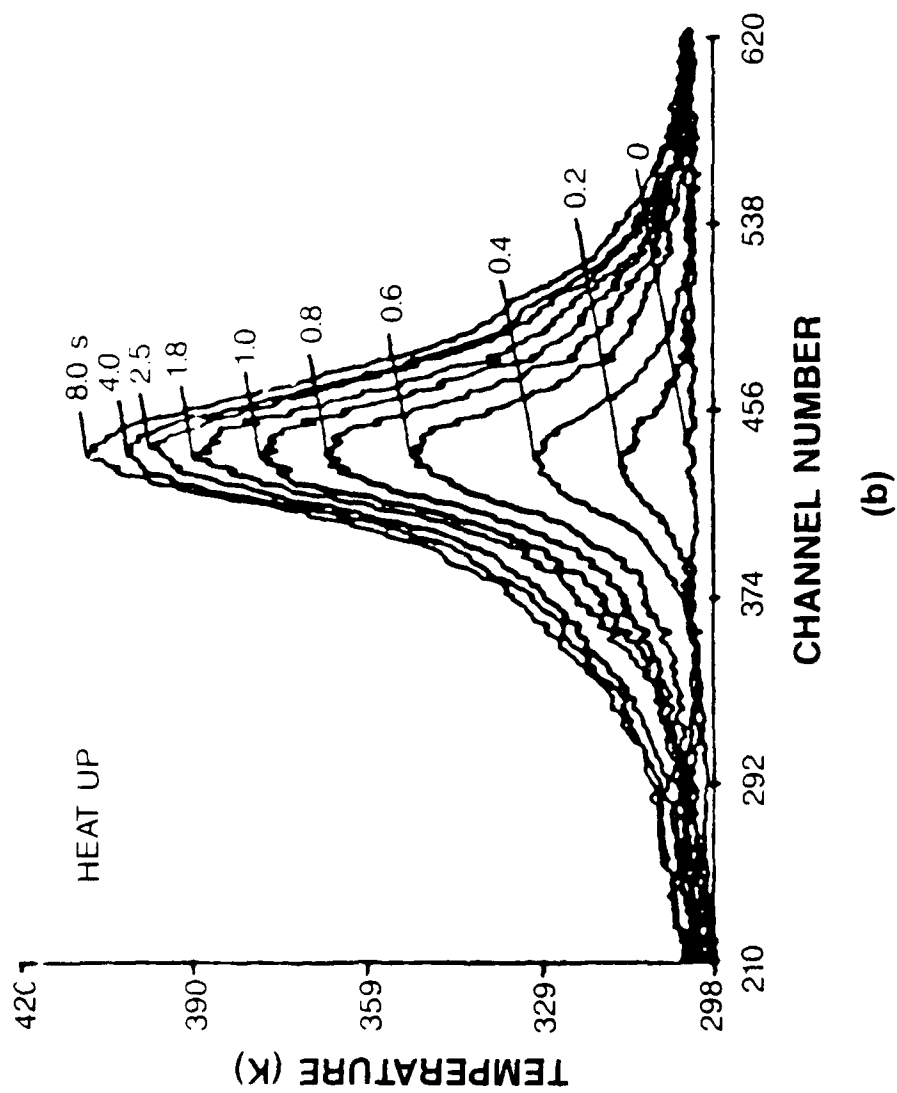


Figure 5(b)

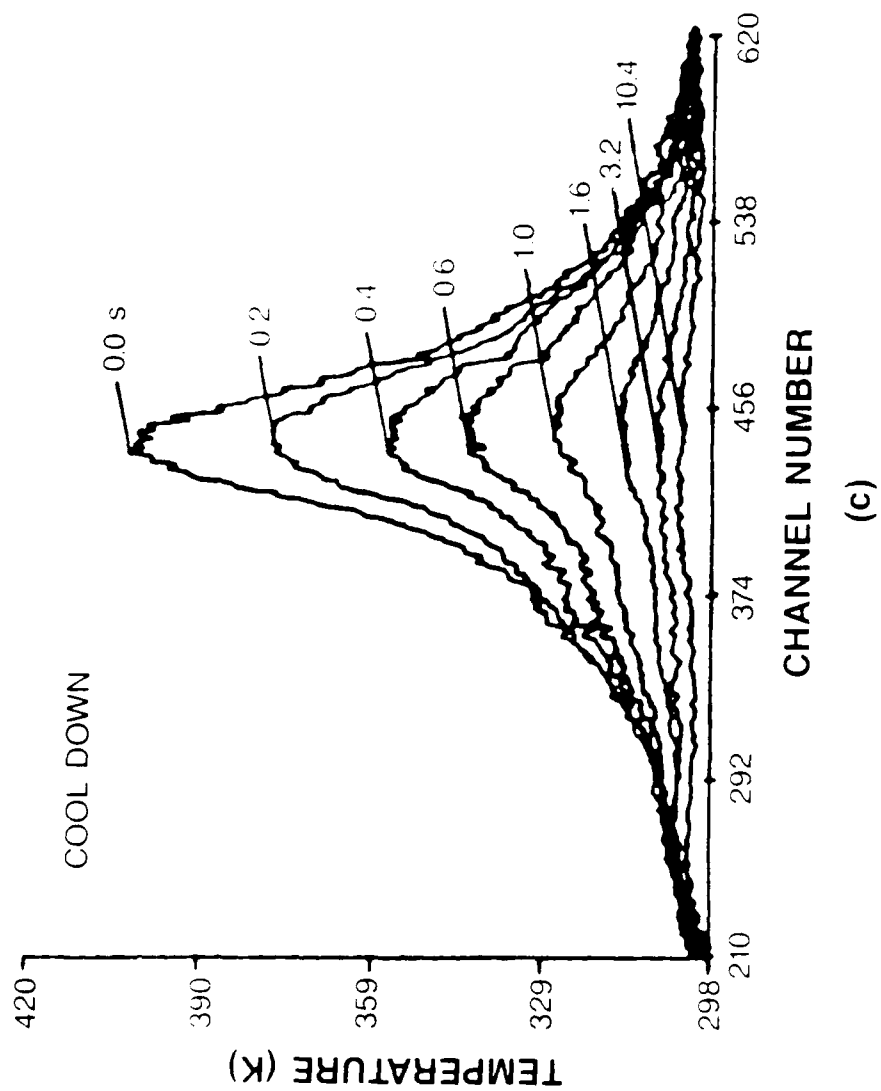


Figure 5(c)

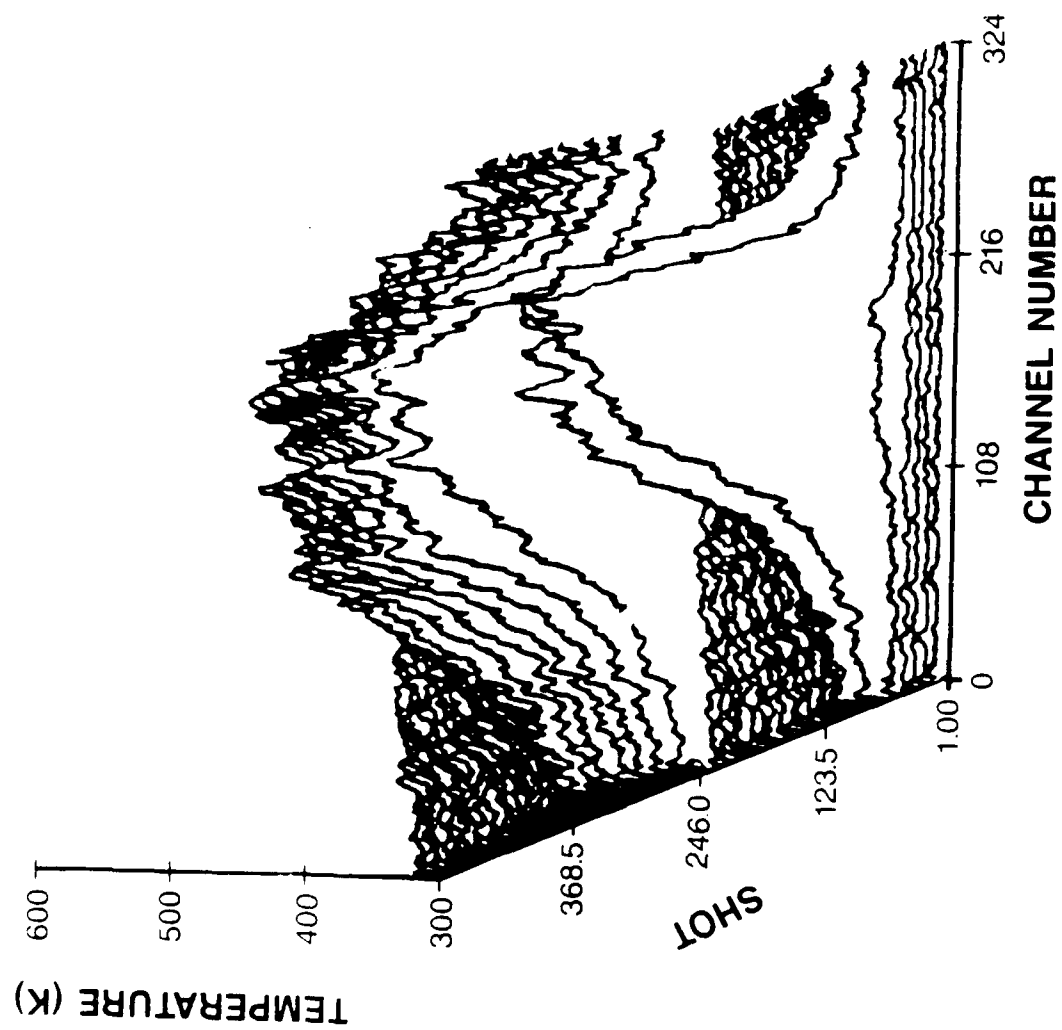


Figure 6

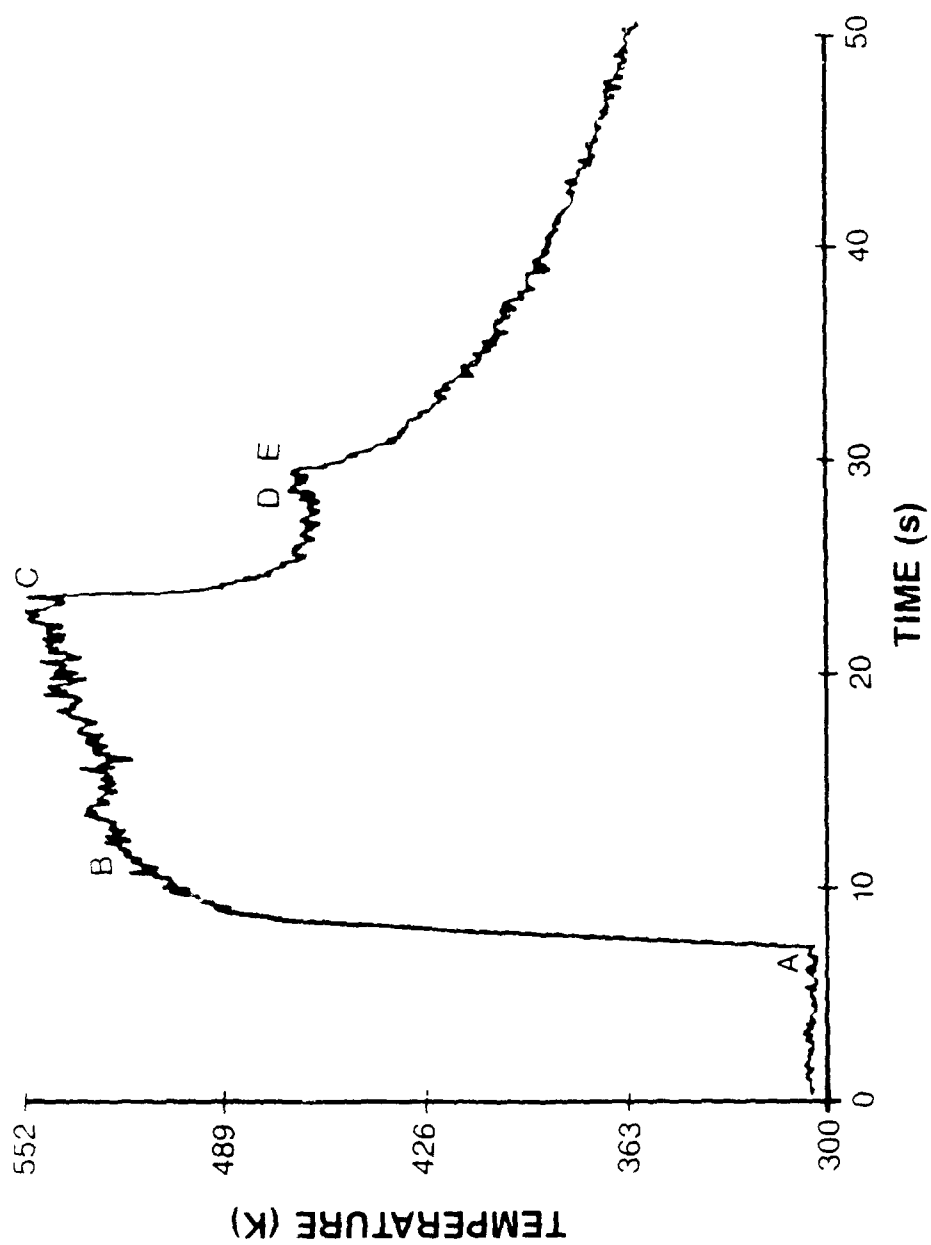


Figure 7